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Indian Standard

METHODS OF CHEMICAL TESTING OF LEATHER

(*First Revision*)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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November 1971

IS : 582 - 1970
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(First Revision)

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CONTENTS

PAGE

0. FOREWORD 4
LC : 0 GENERAL 6
LC : 1 DETERMINATION OF VOLATILE MATTER 9
LC : 2 DETERMINATION OF MOISTURE 11
LC : 3 DETERMINATION OF TOTAL ASH 14
LC : 4 DETERMINATION OF SOLVENT EXTRACTABLE SUBSTANCES 16
LC : 5 DETERMINATION OF NITROGEN AND HIDE SUBSTANCE ... 19
LC : 6 DETERMINATION OF WATER SOLUBLE MATTER 22
LC : 7 DETERMINATION OF SULPHATED ASH OF WATER SOLUBLES 24
LC : 8 DETERMINATION OF WATER INSOLUBLE ASH 25
LC : 9 DETERMINATION OF WATER SOLUBLE ORGANIC SUBSTANCES 26
LC : 10 DETERMINATION OF CHROMIC OXIDE CONTENT ... 27
LC : 11 DETERMINATION OF ALUMINIUM AS ALUMINA (Al_2O_3) IN ALUM TANNED LEATHER 31
LC : 12 DETERMINATION OF ZIRCONIUM 36
LC : 13 DETECTION OF PRESENCE OF FORMALDEHYDE 39
LC : 14 DETERMINATION OF FREE FORMALDEHYDE 40
LC : 15 DETERMINATION OF COMBINED OIL 41
LC : 16 DETERMINATION OF WATER SOLUBLE MAGNESIUM SALTS (EPSOM SALT) 42
LC : 17 DETERMINATION OF GLUCOSE 45
LC : 18 DETERMINATION OF *p*H AND DIFFERENCE FIGURE OF WATER SOLUBLE MATTER 50
LC : 19 DETERMINATION OF TOTAL CHLORIDES 53
LC : 20 DETERMINATION OF TOTAL SULPHATES 54
LC : 21 DETERMINATION OF BOUND ORGANIC SUBSTANCES AND DEGREE OF TANNAGE 55
LC : 22 TEST FOR CORROSION PRODUCED BY LEATHER IN CONTACT WITH METAL 57
LC : 23 DETERMINATION OF SILICONE 60
LC : 24 DETERMINATION OF IRON 66
LC : 25 DETERMINATION OF COPPER 68
LC : 26 DETERMINATION OF PHOSPHORUS 69

Indian Standard

METHODS OF CHEMICAL TESTING OF LEATHER

(First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 16 July 1970, after the draft finalized by the Leather Sectional Committee had been approved by the Chemical Division Council.

0.2 This standard is the amalgamated revision of all chemical methods of test specified in IS : 582-1954* and IS : 1016-1956†. These methods were originally based on BS 1309 : 1946 'Sampling and analysis of vegetable tanned and chrome tanned leathers', and 'Official methods of sampling and tests (1954)', adopted by the Society of Leather Trades Chemists (SLTC), UK. Since the publication of these standards, newer types of tannage have been introduced as well as traditional methods of tannage have been changed in the field of leather in this country. Later, British Standards Institution and SLTC have also revised their standards on methods of test; International Union of Leather Chemists' Societies (IULCS) which came into existence in the meantime, has from time to time published a number of standards on testing of leather. A technical committee set up under the aegis of International Organization for Standardization, ISO/TC 120 Leather, in 1966 has in principle decided to base all its standards on methods published by SLTC and IULCS. Consequent on these developments, the Committee responsible for the preparation of this standard decided to revise IS : 582-1954* and IS : 1016-1956† adopting methods wherever possible from the corresponding methods by SLTC and IULCS. Certain new methods have been introduced in view of the formulations of certain material specifications for leathers of special tannages.

0.3 This standard contains only chemical methods of test for leather. In the formulation of this standard, considerable assistance has been drawn from the following publications:

---

*Methods of sampling and test for vegetable and chrome tanned leathers.

†Methods of sampling and test for oil tanned leathers (*since withdrawn*).

ASTM Part XV : 1969 Paper : Packaging : Cellulose : Casein : Flexible Barrier Materials : Leather. American Society for Testing and Materials, USA.

BS 1309-1959 Methods of sampling and analysis of vegetable tanned and chrome tanned leathers. British Standards Institution, UK.

ALCA Methods of analysis. American Leather Chemists' Association, USA.

Official methods of analysis. Society of Leather Trades' Chemists, UK.

0.4 In this standard, in order to compare components of similar types of leather, it has been prescribed to calculate all results on zero percent volatile matter basis instead of 14 percent moisture basis, as given in the original standards.

0.5 In reporting the result of a test made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960*.

---

*Rules for rounding off numerical values (*revised*).

GENERAL

[LC : 0]

1. SCOPE

1.1 This standard prescribes the methods of chemical testing for all types of leathers.

1.2 Should there be any inconsistency between the methods prescribed in this standard and those given in the individual material specification, then the latter shall prevail.

2. TERMINOLOGY

2.1 For the purpose of this standard, definitions given in IS : 1640-1960* shall apply, in addition to those given in **2.1.1** to **2.1.9**.

2.1.1 *Bound Organic Substances* — The difference between 100 and the sun of the values of the percentage of volatile matter, water insoluble ash, extractable substances, hide substances and water soluble organic substances in a leather. It is expressed as percent by weight of a leather.

2.1.2 *Chromic Oxide Content of Leather* — The quantity of chromium compounds found in leather, calculated as chromic oxide.

2.1.3 *Difference Figure* — The difference between the *p*H value of a solution and its ten-fold dilution. It is a measure for the strength of acids and bases. This value will never exceed the value 1.

2.1.4 *Extractable Substance* — Substances (fats and other solubles) which it is possible to extract from leather with dichloromethane or petroleum hydrocarbon solvent 40/60.

2.1.5 *Total Ash* — The residue left from burning leather in an open crucible at 800±25°C after sulphating.

2.1.6 *Volatile Matter Content* — Volatile matter content is defined as the weight lost by the leather when dried at 102±2°C to constant weight.

2.1.7 *Water Soluble Matter* — Quantity of all those substances which under certain conditions are leached out of the leather by water. These are principally organic tannins, non-tannins and mineral salts.

2.1.8 *Water Soluble Inorganic Substances* — Sulphated ash of water soluble matter.

---

*Glossary of terms relating to hides, skins and leather.

2.1.9 *Water Soluble Organic Substances* — The difference between the water soluble matter and the water soluble inorganic substances.

3. QUALITY OF REAGENTS

3.1 Unless specified otherwise, 'pure chemicals' and distilled water (*see* IS : 1070-1960*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. EXPRESSION OF TEST RESULTS

4.1 Unless otherwise agreed to between the purchaser and the supplier, all results of test shall be expressed on zero percent volatile matter.

4.2 In complete leather analysis, the result shall be determined to one decimal place. For vegetable- and chrome-tanned leathers, they may be represented as in the following examples:

Examples:

a) *For a vegetable-tanned leather*

	ACTUAL COMPOSITION	COMPOSITION CALCULATED ON LEATHER WITH ZERO PERCENT VOLATILE MATTER
Hide substance	*	*
Bound organic substances	*	*
Leather substance	*	*
Water soluble organic substances	*	*
Sulphated ash of water solubles	*	*
Water insoluble ash	*	*
Substances extractable with solvent (fats and other soluble substances)	*	*
Total ash, sulphated	*	*
Volatile matter	*	*
Degree of tannage	†	†
*p*H value of the aqueous leather extract	†	†
*p*H difference figure	†	†

NOTE — The result should be indicated in 'percent' where the sign (*) appears and in 'exact number' where the sign (†) appears.

---

*Specification for water, distilled quality (*revised*).

b) *For chrome-tanned leather*

	ACTUAL COMPOSITION	COMPOSITION CALCULATED ON LEATHER WITH ZERO PERCENT VOLATILE MATTER
Total ash, sulphated	*	*
Chromic oxide, Cr_2O_3	*	*
Sulphated ash of water solubles	*	*
Substances extractable with dichloromethane or petroleum hydrocarbon solvents 40/60 (fats and other soluble substances)	*	*
Volatile matter	*	*
Hide substance	*	*
*p*H value of aqueous leather extract	†	†
*p*H difference figure	†	†

NOTE — The result should be indicated in 'percent' where the sign (*) appears and in 'exact number' where the sign (†) appears.

DETERMINATION OF VOLATILE MATTER

[LC : 1]

(*This method supersedes that in* **6** *of IS: 582-1954**)

1. SCOPE

1.1 This method prescribes a procedure for the determination of volatile matter content in all types of leather.

2. OUTLINE OF THE METHOD

2.1 A known quantity of leather is dried at 102 ± 2°C to constant weight and the weight lost by the leather is expressed as a percentage on the undried sample of the leather.

NOTE — An exact determination of moisture content in the leather is not possible. At elevated temperatures other volatile substances escape and tannins and fats may get oxidized. Some adsorbant water may be left in the leather after drying.

3. APPARATUS

3.1 Desiccator

3.2 Oven — set to 102 ± 2°C.

3.3 Weighing Bottles and Dishes — flat, shallow, weighing bottle with ground-in stopper, or flat, open dishes (*see* IS : 1574-1960†).

NOTE — For more accurate work, use weighing bottles with ground-in stoppers.

4. PROCEDURE

4.1 Sample the leather and prepare a test portion for chemical testing in accordance with IS : 5868-1969‡. Weigh approximately 3 g of the ground leather from the test portion to an accuracy of 0·001, and dry in an oven at 102±2°C for five hours to constant weight. With a normal oven, five hours drying period should be sufficient. Weigh after cooling in the desiccator for half an hour. Check the weight after a further one-hour of drying and half an hour of cooling. If, after redrying, there is a further reduction in weight of more than 3 mg (0·1 percent of the original weight), continue drying but not for longer than eight hours in all.

NOTE 1 — Small glass weighing bottles with ground-in stoppers allow more accurate work than open dishes. Only one should be put into a small desiccator to cool at a time and at the most two in a large desiccator.

---

*Methods of sampling and test for vegetable and chrome tanned leathers.

†Specification for glass weighing bottles.

‡Methods of sampling for leather.

NOTE 2 — For curried and stuffed leathers accurately weighed material shall be first freed from fats and other solubles by solvent extraction by Soxhlet extraction method. The extracted material is then freed from the solvent and then dried as in **4.1**. The total loss of weight represents the sum of the volatile matter, fats and other solubles. The total loss of weight less the quantity of fats and other solubles gives the volatile matter content.

5. CALCULATION

5.1 Calculate the volatile matter, percent by weight, as follows:

$$\text{Volatile matter, percent by weight} = \frac{W_1 - W_2}{W_1} \times 100$$

where

W_1 = weight of the material in g before drying, and
W_2 = weight of the material in g after drying.

5.2 Permissible Error — The result of a duplicate determination shall not differ by more than 0·2 percent calculated on original weight.

6. REPORT

6.1 The report shall state the following:

a) Any deviation from the method described in **4.1** (*see* Notes 1 and 2 in **4.1**), and
b) Mean value of the determinations in accordance with **5.1**.

DETERMINATION OF MOISTURE

[LC : 2]

1. SCOPE

1.1 This method prescribes a procedure for the rapid determination of moisture in pelts and wet leather.

2. OUTLINE OF THE METHOD

2.1 A specified amount of leather or pelt is treated with toluene and the moisture along with toluene is distilled off and collected in the receiver or trap. Toluene being lighter fraction, separates out at the top and from the graduated scale, moisture is determined. Finally the percentage of moisture is calculated by reckoning it back to the actual amount of leather taken for the determination.

3. APPARATUS

3.1 Assembly — The assembly of the apparatus consists of a glass flask heated by suitable means and provided with a reflux condenser discharging into a trap and connected to the flask. The connections between the trap and the condenser and the flask should be of interchangeable ground glass joints (*see* IS : 5165-1969*). The trap serves to collect and measure the condensed water, and to return the solvent to the flask. The assembly of the apparatus is shown in Fig. 1 and the various components are described in **3.1.1** to **3.1.4**.

3.1.1 *Flask* — a 500-ml flask of the shape shown in Fig. 1, made of hard alkali resistant glass, well-annealed and as free as possible from striae and similar defects.

3.1.2 *Condenser* — a water cooled, glass reflux condenser, having a jacket approximately 400 mm long and an inner tube of 9·5 to 12·7 mm outside diameter. The tip of the condenser to be inserted in the trap may be ground off at an angle of 30° from the vertical axis of the condenser. When inserted into the trap, the tip of the condenser shall be 6 to 7 mm above the surface of the liquid in the trap after distillation conditions have been established.

3.1.3 *Receiver* — otherwise called the trap, made of hard alkali resistant glass, well-annealed and as far as possible free from striae and similar defects, provided with ground glass joints, with the shape, dimensions and tolerances given in Fig. 1; consisting essentially of the upper chamber, together with the tube and ground joint leading to the flask, and the graduated tube. The graduated portion shall have a capacity of 5 ml when filled to the highest graduation mark. The scale shall cover the range of 0 to 5 ml and shall be graduated at intervals of 0·1 ml. The graduation

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*Specification for interchangeable conical ground-glass joints.

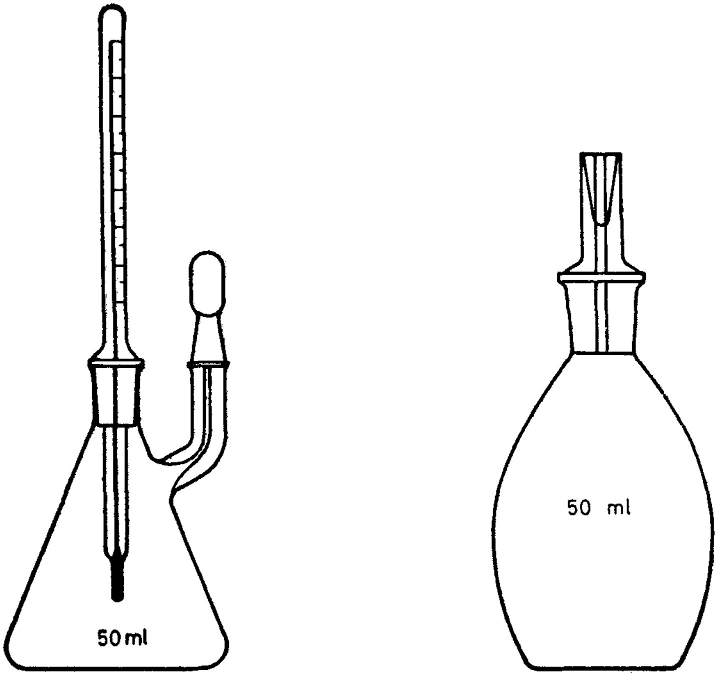

Fig. 1 Moisture Distillation Apparatus

marks corresponding to each millilitre shall be numbered and carried completely round the tube. The graduation marks midway between the numbered marks shall be carried three-quarters-way round the tube and the remaining marks shall be carried half-way round the tube. The capacity of the width occupied by each graduation mark shall not exceed 0·05 ml.

3.1.4 *Heat Source* — The source of heat may be either an oil-bath or an electric heater provided with a sliding rheostat or other means of heat control. The temperature of the oil in the bath should not be very much higher than the boiling point of toluene.

3.2 Copper Wire — long enough to extend through the condenser, with one end twisted into a spiral. The diameter of the spiral should be such that it fits snugly within the graduated portion of the receiver and yet capable of being moved up and down.

4. REAGENTS

4.1 Potassium Dichromate-Sulphuric Acid Cleaning Solution

4.2 Toluene — Saturate the toluene by shaking with a small quantity of water and distil. Use the distillate for moisture determinations.

5. PROCEDURE

5.1 Clean the entire apparatus with potassium dichromate-sulphuric acid cleaning solution to minimize the adherence of water droplets to the sides of the condenser and the receiver. Rinse thoroughly with water and dry completely before using. The quantity of material taken for the test is determined by the amount of moisture present (*w/w*). Normally 20 g of the pelt or leather cut into square pieces of about 3-mm side should be sufficient.

5.2 Place the required quantity of material, accurately weighed, in the distillation flask and add an equal volume of toluene, as desired but never less than 10 ml and swirl to mix. Assemble the apparatus and fill the receiver with the solvent by pouring it through the condenser until it begins to overflow into the distillation flask. Insert a loose cotton plug in the top of the condenser to prevent condensation of atmospheric moisture within the tube. In order that the refluxing may be under control, wrap the flask and tube leading to the receiver with asbestos cloth. Heat the flask so that the distillation rate is about 100 drops per minute. When the greater part of the water has distilled over, increase the distillation rate to about 200 drops per minute and continue until no more water is collected. Purge the reflux condenser occasionally during the distillation with 6-ml portions of toluene to wash down and moisture adhering to the wall of the condenser. The water in the receiver may be made to separate from the toluene by moving the spiral copper wire up and down in the condenser and receiver occasionally, thus causing the water to settle at the bottom of the receiver. Reflux until the water level in the receiver remains unchanged for 30 minutes and then shut off the source of heat. Flush the condenser with toluene, as required, making use of the spiral copper wire to discharge any moisture droplets. Immerse the receiver in water at about 27°C for at least 15 minutes or until the toluene layer is clear, and then read the volume of water.

NOTE — The distillation method followed here employs Dean and Stark apparatus and a hydrocarbon solvent as the carrier. The test may also be carried out in Gisiger apparatus using tetrachloro-ethane ($CHCl_2$-$CHCl_2$) as the carrier.

6. CALCULATION

6.1 Calculate the moisture content as follows:

$$\text{Moisture content, percent by weight} = \frac{100\,V D}{W}$$

where

V = volume in ml of water,
D = relative density of water at the temperature at which the volume of water is read, and
W = weight in g of the material taken for the test.

6.1.1 The determination, when done in duplicate, shall not differ by more than 2 percent.

DETERMINATION OF TOTAL ASH

[LC : 3]

(*This method supersedes that in 7 of IS : 582-1954**)

1. SCOPE

1.1 This method prescribes a procedure for the determination of total ash in all types of leather. Ammonium salts are not included.

2. OUTLINE OF THE METHOD

2.1 A known quantity of the leather is burnt at 800±25°C and the residue is sulphated.

2.1.1 The method is inaccurate by the extent to which the leather contains organic metal compounds like silicone. The amount of mineral substances found by ashing may differ from the actual content owing to decomposition, reduction and escape of certain salts.

2.1.2 By treating the ash with sulphuric acid, the salts and oxides are converted into sulphate, but with some salts they shall again be transformed into oxides at the selected temperature of ignition.

3. APPARATUS

3.1 Crucible — platinum or silica crucible.

3.2 Desiccator

3.3 Muffle Furnace — fitted with pyrometer and having thermostatic control.

4. REAGENTS

4.1 Sulphuric Acid — 2 N (*see* IS : 266-1961†).

4.2 Ammonium Nitrate Solution — 10 percent solution (*w/v*).

5. PROCEDURE

5.1 Sample the leather and prepare a test portion for chemical testing in accordance with IS : 5868 - 1969‡. Weigh about 5 g from the test portion to an accuracy of 0·001 g and carbonize it over a low flame in a crucible, that has previously been heated to 800±25°C, cooled and weighed, so that the leather burns with a small flame. Carbonize, particularly curried leather, carefully so that the greases burn slowly. Thoroughly moisten the ash with sulphuric acid and heat over a low flame until sulphuric acid fumes are no longer visible. Then ignite in the muffle furnace at 800±25°C. If carbon-free residue is not obtained in spite of heating at 800±25°C, moisten the residue with a little of ammonium nitrate solution and make it red hot again until it is free from carbon. If complete ashing is not possible even with the help of ammonium nitrate extract, the contents of the crucible with hot water and filter through an ash-free filter paper. Ash the carbon residue with filter paper, add the filtrate to the content of the crucible, evaporate on the water-bath; again heat at 800±25°C until the last traces of visible carbon are removed. Cool in a desiccator and weigh. Repeat heating, cooling and weighing until the weight of the residue is constant.

---

*Methods of sampling and test for vegetable and chrome tanned leathers.

†Specification for sulphuric acid (*revised*).

‡Methods of sampling for leather.

NOTE 1 — For the determination of ash, the dried leather obtained from the determination of volatile matter in accordance with LC : 1 can be used in all cases.

NOTE 2 — It is advisable to extract silicone impregnated leather with the solvent before determining the ash.

NOTE 3 — The ash may be used to determine its constituents of chrome oxide, aluminium oxide or other cations of mineral filler substances.

6. CALCULATION

6.1 Calculate the total ash as follows:

$$\text{Total ash, percent by weight} = \frac{W_1}{W_2} \times 100$$

where

W_1 = weight in g of the sulphated ash, and

W_2 = weight in g of the material taken for the test.

DETERMINATION OF SOLVENT EXTRACTABLE SUBSTANCES

[LC : 4]

(*This method supersedes that in* 8 *of IS : 582-1954**)

1. SCOPE

1.1 This method is intended for the determination of substances extractable in dichloromethane or petroleum hydrocarbon solvent 40/60 in all types of leather.

2. OUTLINE OF THE METHOD

2.1 A known quantity of leather is extracted with a specified solvent in a Soxhlet apparatus, and the fat and other solubles extracted are dried, weighed and expressed as percent on the leather.

2.1.1 Dichloromethane or other organic solvents may not extract all fatty and similar substances. They may be partly soluble and partly bound to the leather. The solvent may dissolve non-fatty substances like sulphur and impregnates, both of which cause difficulty in the determination of the acid value and saponification value of fat.

3. APPARATUS

3.1 Desiccator

3.2 Filter Paper Thimbles or Glass Filter Bells†

3.3 Oven — to maintain temperature at 102±2°C.

3.4 Soxhlet Extraction Apparatus — with extraction flasks of suitable size.

4. REAGENTS

4.1 Dichloromethane (CH_2Cl_2) — boiling point 38 to 40°C, freshly distilled, kept in dark flask over calcium oxide (CaO).

Note 1 — Dichloromethane has toxic properties and should be used with caution.

---

*Methods of sampling and test for vegetable and chrome tanned leathers.

†Shleicher and Schull, No. 603 Whatman extraction thimbles 33×80 or equivalent are suitable.

NOTE 2 — Dichloromethane that has stood for a long time should be tested for the presence of hydrochloric acid which may possibly have formed, as follows:

> Shake 10 ml of dichloromethane with 1 ml of 0·1 N silver nitrate solution. If the silver nitrate solution becomes turbid, the solvent should be distilled and kept in the dark over calcium oxide (CaO).

NOTE 3 — Dichloromethane which has been used once may be used again after distillation.

4.2 Petroleum Hydrocarbon Solvent 40/60 — *see* IS : 1745-1966*.

5. PROCEDURE

5.1 Sample the leather and prepare a test portion for chemical testing in accordance with IS : 5868-1969†. Dry the extraction flask with two or three glass beads in it by heating for half an hour at 102±2°C; weigh after cooling in a desiccator. Weigh accurately about 10·0±0·1 g of leather from the test portion. Place it in a Soxhlet extractor with the tared flask and press the leather evenly into the filter paper thimble or glass bell. Cover the leather with a thin layer of grease-free cotton wool. Start extraction of the solubles with the specified solvent and after at least 30 changes of solvent, distil the solvent from the flask, containing the extract. Dry the extract in the oven for 4 h at 102±2°C, if drops of water are visible, with the addition of 1 to 2 ml ethyl alcohol; weigh after cooling for 30 minutes in a desiccator. Check the weight twice after further drying for 1 h and cooling for 30 minutes. If redrying results in a further weight reduction of more than 10 mg, continue drying up to a maximum of 8 h. Preserve the extracted leather for other tests under LC : 6.

NOTE 1 — The particular solvent to be used should be specified in the material specified giving reference to this method.

NOTE 2 — Use of a current of air to free the fat from the bulk of the solvent before drying in the oven facilitates the process.

NOTE 3 — Dichloromethane or petroleum hydrocarbon solvent may also dissolve non-fats like sulphur. As sulphur causes difficulty in the determination of acid value and saponification value of fat, if these values of the extracted fat are to be determined, sulphur should be removed in the following way:

> The presence of sulphur is recognizable by the yellow precipitation in the flask. Dissolve the extract in the smallest possible quantity of diethyl ether [$(C_2H_5)_2O$] and filter through a little wadding into a previously weighed flask. After thoroughly washing out the wadding filter with ether, remove the ether from the extract in the flask by distillation. If sulphur should again precipitate, repeat the procedure. After the diethyl ether has been distilled off, dry the flask with the residue again and weigh.

NOTE 4 — If sulphur determination is desired, the same may be expressed as the difference between the extractable substances (*see* **6.1**) and the value obtained following the procedure given under Note 3.

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*Specification for petroleum hydrocarbon solvents (*first revision*).

†Methods of sampling for leather.

6. CALCULATION

6.1 Calculate the extractable substances in leather as follows:

$$\text{Extractable substances, percent by weight} = 100 \times \frac{W_2}{W_1}$$

where

W_2 = weight in g of the extract, and
W_1 = weight in g of the material taken for the test.

6.2 Permissible Error — The results of a duplicate determination shall not differ from one another by more than 0·2 percent calculated on the original weight of the material taken for the test.

7. REPORT

7.1 The report shall contain the following:

a) Reference to the method;
b) Deviation from this method, if any; and
c) Mean value of the results (*see* **6.1** and **6.2**).

DETERMINATION OF NITROGEN AND HIDE SUBSTANCE

[LC : 5]

(*This method supersedes that in* **16** *of IS : 582-1954**)

1. SCOPE

1.1 This method prescribes a procedure for the determination of the total amino nitrogen and its conversion to hide substances in any type of leather.

2. OUTLINE OF THE METHOD

2.1 The nitrogenous substances present in the leather are converted to ammonium sulphate by digestion with concentrated sulphuric acid in the presence of a catalyst by the Kjeldahl method. The ammonia is liberated by the addition of excess of caustic soda, distilled into an absorbent solution and estimated by titration using mixed indicator. From this, percentage of nitrogen and hide substances are calculated.

2.1.1 The determination of hide substance from nitrogen content is based on the fact that the grease and ash-free dry substance of the pelts of various animals has a somewhat different, but practically constant nitrogen content for certain types of animals. Other nitrogenous substances, such as fixatives, dyestuffs, syntans and cationic fats falsify the value for the hide substance, when it is not possible to obtain an accurate result for the hide substances.

3. APPARATUS

3.1 Distillation Apparatus

3.1.1 *Kjeldahl Flask* — 200 to 300 ml capacity if an external source of steam is to be used for the distillation or 700 ml capacity if the steam is to be liberated within the distillation system.

3.1.2 *Receiver* — 300 to 400 ml capacity.

4. REAGENTS

4.1 Fuming [7 Percent Free Sulphur Trioxide (SO_3)] or Concentrated Sulphuric Acid — relative density 1·84 (*see* IS : 266-1961†).

4.2 Catalyst Mixture — catalyst mixture of 100 mg of anhydrous copper sulphate and 6 to 8 g of potassium sulphate.

Note — Any other suitable catalyst mixture, which considerably shortens the digestion period, may be used.

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*Methods of sampling and test for vegetable and chrome tanned leathers.

†Specification for sulphuric acid (*revised*).

4.3 Phenolphthalein Indicator — Dissolve 1 g of phenolphthalein in 100 ml of ethyl alcohol (50 percent by volume) (*see* IS : 2263-1962*).

4.4 Sodium Hydroxide Solution — 35 percent (*w*/*v*).

4.5 Saturated Solution of Borate-Free Boric Acid

4.6 Indicator Solution — The indicator solution prepared in the following way is found suitable. Mix indicator solutions of 0·06 percent methyl red and 0·04 percent methylene blue in 96 percent ethyl alcohol. Take 2 ml of the mixed indicator solution in one litre of water.

4.7 Standard Sulphuric or Hydrochloric Acid — approximately 0·5 N.

5. PROCEDURE

5.1 Sample the leathers and prepare a test portion for chemical tests in accordance with IS : 5868-1969†. Weigh to an accuracy of 0·000 1 g about 3 g from the test portion and place in Kjeldahl flask. Add 30 ml of fuming or concentrated sulphuric acid and 5 g of catalyst mixture. Heat to boiling, first with a low and later with a high flame, until 1 h after all carbon has been oxidized. Place a small funnel in the neck of the flask to avoid undue loss of acid during digestion. If an external source of steam is to be used, cool and digest, dilute with approximately 50 ml of distilled water; cool and transfer to the distilling flask. Rinse the Kjeldahl flask twice with 20 ml of distilled water each time. If necessary, dilute further with additional distilled water, add a few drops of phenolphthalein and make the solution alkaline with an excess 35 percent caustic soda solution (approximately 60 ml) and steam distil. Alternatively, if the digestion is to be made in Kjeldahl flask, allow the flask and the contents to cool. After cooling, dilute with 250 ml of water; add a few fragments of anti-bump material (for example, ignited porous earthenware) and 1 ml of phenolphthalein solution. Pour excess of sodium hydroxide solution (approximately 70 ml) carefully down the side of the flask. Next connect the flask with a vertical condenser by means of a tube bent twice preferably including a spray trap. Distil the ammonia with water vapour into a receiver of 300 to 400 ml capacity containing 100 ml of saturated boric acid and indicator solution. Dip the cooling tube into the boric acid. The distilled ammonia colours the indicator green. Distil until there is 150 to 200 ml of distillate. Before ending the distillation, lower the receiver so that the cooling tube no longer dips in. Distil for another 3 minutes; rinse the end of the condenser with distilled water. Titrate the ammonia with standard sulphuric acid to *p*H 4·6. When using the indicator (*see* **4.6**) titrate to the first constant pale pink colour.

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*Methods of preparation of indicator solutions for volumetric analysis.

†Methods of sampling for leather.

NOTE 1 — The purity of the chemicals used should be tested by carrying out a blank experiment with pure cane sugar.

NOTE 2 — Leather containing more than 5 percent grease should be approximately degreased before the Kjeldahl digestion. This is done by soaking the weighed sample *in petroleum hydrocarbon solvent* 40/60 (*see* IS : 1745-1966*) for a short time and then discarding the solvent and finally allowing the leather to dry in the air.

NOTE 3 — The end point of the titration is approximately *p*H 4·6. Potentiometric and other indicator systems known to give an accurate colour change at *p*H 4·6 may also be used.

NOTE 4 — Slight modification of the apparatus for distilling, such as running the 35 percent sodium hydroxide from a tap funnel and collecting the distillate in a flask with a guard tube through which boric acid may be introduced, is allowed.

NOTE 5 — The determination may also be made with 0·5 g of leather weighed to 0·000 2 g, 15 to 20 ml concentrated or fuming sulphuric acid and 2·5 g catalyst. Titration of ammonia is then done with 0·1 N acid.

6. CALCULATION

6.1 Calculate the nitrogen content as follows:

$$\text{Nitrogen content, percent by weight} = \frac{1{\cdot}4 \times V \times N}{W}$$

where

V = volume in ml of acid used in the titration (after correction for the blank value),

N = normality of the sulphuric or hydrochloric acid, and

W = weight in g of the material taken for the test.

6.2 Calculate the hide substance as follows:

$$\text{Hide substance, percent by weight} = 5{\cdot}62\ X$$

where

X = nitrogen content, percent by weight (**6.1**).

6.3 Permissible Error — With a duplicate determination, the result shall not differ from one another by more than 0·1 percent of nitrogen content.

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*Specification for petroleum hydrocarbon solvents (*first revision*).

DETERMINATION OF WATER SOLUBLE MATTER

[LC : 6]

(*This method supersedes that in* **10** *of IS : 582-1954**)

1. SCOPE

1.1 This method prescribes a procedure for the determination of water soluble organic and inorganic substances in all types of leather.

2. OUTLINE OF THE METHOD

2.1 A specified amount of leather is shaken with water in a shaker under the specified condition. The dissolved substances are then evaporated and from the residue, the percentage of the water soluble matter on the original weight of the leather is calculated.

3. APPARATUS

3.1 Desiccator

3.2 Evaporating Basin — quartz, porcelain, or platinum with flat bottom to hold 50 ml of liquid.

3.3 Flask — 650 to 750 ml capacity, wide neck, and having close-fitting glass or rubber stopper preferably a vacuum thermos flask.

3.4 Oven — set at 102±2°C.

3.5 Shaker — suitable mechanical shaker with 50±10 rev/min.

4. PROCEDURE

4.1 Take the leathers after extraction from the Soxhlet extractor (*see* **5.1** of LC : 4). Alternatively, sample the leather and prepare a test portion for chemical testing in accordance with IS : 5868-1969†. Accurately weigh 10·00 g of leather from the test portion. Remove the grease completely from leather, following the procedure given in LC : 4. Free the extracted leather from solvent by spreading it out on a clean surface and leaving it exposed to the air, but do not heat on a water-bath or in a hot-oven. Transfer the grease-free leather to a flask (*see* **3.3**). Add 500 ml of water and shake in a mechanical shaker at 27±2°C for 2 h. Separate the liquid from the residual leather by filtration using a Whatman No. 11 or its equivalent filter paper and reject the first 50 ml of the filtrate. Evaporate

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*Methods of sampling and test for vegetable and chrome tanned leathers.

†Methods of sampling for leather.

50 ml of the filtrate to dryness in an evaporating basin, which has been previously heated to 800±25°C, cooled and weighed, on a hot-water-bath. Dry this residue at 102±2°C in an oven for approximately 2 h. Cool in a desiccator for 20 minutes and weigh quickly. Only one dish should be put in a small desiccator and at the most two in a large desiccator. Return to the oven for a further period of 1 h, cool in a desiccator for 20 minutes and reweigh. Repeat this process of drying until the decrease in weight between two successive weighings is less than 2 mg, but not more than 8 h. Reserve the rest of the filtrate and the residue for test under LC : 7.

NOTE 1 — The result obtained for the content of water soluble matter of a leather depends on the extent to which the leather is ground, the temperature, the extraction period and the ratio of leather to water. To obtain comparable results, it is consequently imperative that the test conditions should be accurately observed.

NOTE 2 — If the prescribed extraction temperature of 27±2°C is not possible to be maintained in the test room, it is advisable to use a thermos flask. The range of values is likely to differ by about 0·5 percent over the permitted temperature range.

NOTE 3 — If the quantity of water soluble mineral substances is likely to be less than 1·0 percent of the leather weight, the determination should be made on a 100- or 200-ml aliquot.

5. CALCULATION

5.1 Calculate the total water soluble matter as follows:

$$\text{Total water soluble matter, percent by weight} = 100\,W$$

where

W = weight in g of the dry residue left after drying.

5.2 Permissible Error — The results of a duplicate determination shall not differ from one another by more than 0·2 percent calculated on the original weight of the material taken for the test.

DETERMINATION OF SULPHATED ASH OF WATER SOLUBLES

[LC : 7]

1. SCOPE

1.1 This method prescribes a procedure for the determination of water soluble inorganic substances expressed as sulphated ash of water solubles in all types of leather.

2. OUTLINE OF THE METHOD

2.1 A specified amount of leather is shaken with water in a shaker under specified conditions. The dissolved substances are then evaporated. The residue is then treated with sulphuric acid and finally the percentage of the sulphated ash is calculated on the weight of the original material taken.

3. APPARATUS

3.1 Apparatus as in **3** of LC : 6 shall be used.

4. REAGENT

4.1 Sulphuric Acid — approximately 2 N (*see* IS : 266-1961*).

5. PROCEDURE

5.1 Take the residue as obtained under **4.1** of LC : 6; alternatively, evaporate 50 ml of the filtrate as obtained under **4.1** of LC : 6 at 102±2°C until dry. Thoroughly wet the residue in the dish with a few drops of sulphuric acid, fume over a low flame until no sulphuric acid vapour is visible. Heat until red hot, preferably in a muffle furnace at 800±25°C for 15 minutes. Cool in a desiccator and weigh as quickly as possible. Repeat the addition of acid, heating, cooling and weighing until the weight of the residue is constant.

6. CALCULATION

6.1 Calculate the sulphated ash of water soluble matter as follows:

$$\text{Sulphated ash of water soluble matter, percent by weight} = \frac{W_1}{W_2} \times 100$$

where

W_1 = weight in g of the sulphated ash, and

W_2 = weight in g of the prepared material taken before extracting the solvent soluble substances (*see* LC : 4).

6.2 Permissible Error — The results of a duplicate determination shall not differ from one another by more than 0·2 percent calculated on the original weight of the material taken for the test.

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*Specification for sulphuric acid (*revised*).

DETERMINATION OF WATER INSOLUBLE ASH

[LC : 8]

(*These methods supersede that in* **12** *of IS : 582-1954**)

1. SCOPE

1.1 These two methods prescribe the procedure for the determination of insoluble ash of all types of leather.

2. OUTLINE OF THE METHODS

2.1 Method A — Total ash and sulphated ash of the water solubles is determined separately. The difference of the percentage of total ash and sulphated ash of water solubles is taken to get percentage of water insoluble ash.

2.2 Method B — The leather, after the determination of water soluble matter, is directly ignited to get water insoluble ash.

3. PROCEDURE

3.1 Method A — Determine the total ash and the sulphated ash of water solubles following the methods given in LC : 3 and LC : 7 respectively. Take their difference.

3.2 Method B — Air dry the leather remaining after the determination of water soluble matter (LC : 6) and ash it completely in accordance with LC : 3.

4. CALCULATION

4.1 Calculate the water insoluble ash, percent by weight, of Methods **A** and B as given in **4.1.1** and **4.1.2**.

4.1.1 *Method A*

Water insoluble ash, percent by weight = $W_1 - W_2$

where

W_1 = total ash, percent by weight; and
W_2 = sulphated ash of water solubles, percent by weight.

4.1.2 *Method B*

$$\text{Water insoluble ash, percent by weight} = \frac{W_3}{W_4} \times 100$$

where

W_3 = weight in g of sulphated insoluble ash; and
W_4 = weight in g of originally prepared sample of leather taken before extracting the solvent soluble substances (*see* LC : 4).

4.2 Permissible Error — The results of the duplicate determinations shall not differ by more than 0·1 percent on the weight of material taken.

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*Methods of sampling and test for vegetable and chrome tanned leathers.

DETERMINATION OF WATER SOLUBLE ORGANIC SUBSTANCES

[LC : 9]

1. SCOPE

1.1 This method is intended for the determination of water soluble organic substances of all types of leather.

2. OUTLINE OF THE METHOD

2.1 Water solubles and sulphated ash of water solubles of a specific amount of leather are determined. From these two determinations, percentage of water soluble organic substances is calculated.

2.1.1 Any ammonium salts in the filtrate are included as organic substances.

3. PROCEDURE

3.1 Determine the water solubles and sulphated ash of water solubles according to the methods given in LC : 6 and LC : 7 respectively.

4. CALCULATION

4.1 Calculate the water soluble organic substances, as follows:

$$\text{Water soluble organic substances, percent by weight} = W_1 - W_2$$

where

W_1 = water soluble matter, percent by weight; and
W_2 = sulphated ash of water soluble matter, percent by weight.

4.2 Permissible Error — The results of a duplicate determination shall not differ by more than 0·2 percent calculated on the original weight of the material taken.

DETERMINATION OF CHROMIC OXIDE CONTENT

[LC : 10]

(*These Methods supersede that in* **9** *of IS : 582-1954**)

1. SCOPE

1.1 These methods prescribe three procedures for the determination of chromium (as Cr_2O_3) in leathers.

2. METHOD A (WET OXIDATION METHOD)

2.1 Outline of the Method — In this method, the trivalent chromium in the leather is oxidized with an oxidizing mixture containing perchloric acid and sulphuric acid to the hexavalent chromium. Chromic oxide is then determined by iodometric titration of the hexavalent chromium.

2.2 Apparatus

2.2.1 *Kjeldahl or Erlenmeyer Flask* — 300 ml capacity with ground glass stopper.

2.2.2 *Conical Flask* — with ground glass stopper.

2.2.3 *Crucible* — iron or nickel.

2.3 Reagents

2.3.1 *Concentrated Sulphuric Acid* — relative density 1·84 (*see* IS : 266-1961†).

2.3.2 *Perchloric Acid* — 60 to 70 percent (*v/v*).

2.3.3 *Phosphoric Acid* — 85 percent solution (*v/v*).

2.3.4 *Starch Solution* — Triturate 5 g of pure starch and 0·01 g of mercuric iodide with 30 ml of water in a mortar. Pour the resulting paste into one litre of boiling water, boil for 3 minutes, allow the solution to cool and decant off the clear liquid.

2.3.5 *Standard Sodium Thiosulphate Solution* — approximately 0·1 N.

2.3.6 *Potassium Iodide Solution* — approximately 10 percent (*w/v*), iodate-free.

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*Methods of sampling and test for vegetable and chrome tanned leathers.

†Specification for sulphuric acid (*revised*).

2.4 Procedure — Sample the leather and prepare the test portion for chemical testing in accordance with IS : 5868-1969*. Weigh about 1 to 2 g from the test portion and transfer to a heat resistant 300-ml Erlenmeyer or Kjeldahl flask. Add 5 ml of concentrated nitric acid to the sample followed by 15 ml of mixed perchloric and sulphuric acids (5 ml of concentrated sulphuric acid and 10 ml of perchloric acid) and heat to boiling on the wire gauze with a moderate flame. Cover the flask with a funnel so that water may evaporate without loss by splashing. As soon as the reaction mixture begins to turn to orange, lower the flame. After complete change of colour, heat slowly for a further two minutes, cool for a short time in air and then rapidly in cold water, and dilute the contents to approximately 200 ml. To eliminate the chlorine thus created, boil approximately for 10 minutes using earthenware pebbles (glass beads are not efficient to prevent bumping). After renewed cooling, add 15 ml of phosphoric acid to mask any iron present and 20 ml of potassium iodide solution. Leave to stand for 10 minutes in the dark. Then titrate to light green, using 5 ml of a starch solution as indicator, with standard sodium thiosulphate solution.

NOTE 1 — Instead of 60 to 70 percent perchloric acid, 5 to 7 g of ammonium perchlorate may be added for the oxidation.

NOTE 2 — The oxidation of leather with perchloric acid alone should be avoided owing to the acute danger of explosion. The oxidizing acids may be stored as a mixed reagent containing sulphuric and perchloric acids; this will minimize the chance of adding only perchloric acid.

2.5 Calculation

$$\text{Chromium (as } Cr_2O_3 \text{), percent by weight} = \frac{2{\cdot}534\ VN}{W}$$

where

V = volume in ml of standard sodium thiosulphate solution,

N = normality of standard sodium thiosulphate solution, and

W = weight in g of the material taken for the test.

3. METHOD B

3.1 Outline of the Method — In this method, the ash of the ground leather is oxidized by fusion with sodium peroxide, and chromic oxide is determined from the melt by the iodometric titration of the hexavalent chromium.

3.1.1 The ash obtained in LC : 3 is used for the determination of chromic oxide (Cr_2O_3) content which can be determined on the dried leather from the volatile matter determination given in LC : 1. The possibility or error is considerably reduced if these three determinations are carried out with single leather weigh-in.

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*Methods of sampling for leather.

3.2 Apparatus — same as in **2.2**.

3.3 Reagents

3.3.1 *Potassium Carbonate*

3.3.2 *Sodium Tetraborate* — powder.

3.3.3 *Anhydrous Sodium Carbonate* — conforming to IS : 296-1965*.

3.3.4 *Concentrated Hydrochloric Acid* — relative density 1·16 (*see* IS : 265-1962†).

3.3.5 *Potassium Iodide Solution* — approximately 10 percent (*w/v*), iodate-free.

3.3.6 *Standard Sodium Thiosulphate Solution* — approximately 0·1 N.

3.3.7 *Starch Solution* — same as in **2.3.4**.

3.4 Procedure — Transfer the ash as obtained in LC : 3 to a nickel or iron crucible (or alternatively, about 5 g of the ground leather, accurately weighed, may be added directly in the crucible if an accurate value for the ash is not desired) and mix the ash well with the fusion mixture composed of equal parts of soda ash, potassium carbonate and sodium tetraborate, using not less than three nor more than 10 times the weight of the ash. Fuse the mixture gradually and heat to moderate red heat only for one to five minutes. After cooling, place the crucible and melt in a large beaker and pour almost boiling distilled water to dissolve the melt. Cover the beaker with a clock glass to prevent loss by spurting. Transfer the solution to a 500-ml boiling flask. Remove the crucible carefully and wash thoroughly adding the washing to the boiling flask. Filter the solution obtained into an Erlenmeyer flask. Thoroughly wash the filter with hot distilled water. Add a piece of iron (about 3 × 1 cm), freshly cleaned with concentrated hydrochloric acid, to the boiling flask and boil for 10 minutes. Cool the solution and add 10 ml of potassium iodide solution. Replace the stopper of the flask, allow to stand for 2 minutes and then immediately titrate the liberated iodine with standard sodium thiosulphate solution using starch solution as indicator.

3.5 Calculation — same as in **2.5**.

4. METHOD C

4.1 Outline of the Method — In this method the ash of the ground leather is fused with equal parts of anhydrous sodium carbonate, potassium carbonate

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*Specification for sodium carbonate, anhydrous (*revised*).

†Specification for hydrochloric acid (*revised*).

and borax, and chromium is determined from the melt by the iodometric method.

4.1.1 The ash obtained in LC : 3 is used for the determination of chromic oxide (Cr_2O_3) content which may be determined on the dried leather from the volatile matter determination given in LC : 1. The possibility of error is considerably reduced if these determinations are carried out with a single leather weigh-in.

4.2 Apparatus — same as in **2.2.**

4.3 Reagents

4.3.1 *Anhydrous Sodium Carbonate* — conforming to IS : 296-1965*.

4.3.2 *Potassium Chlorate*

4.3.3 *Concentrated Hydrochloric Acid* — relative density 1·16 (*see* IS : 265-1962†).

4.3.4 *Dilute Hydrochloric Acid* — approximately 5 N.

4.3.5 *Ammonium Hydroxide Solution* — relative density 0·90.

4.3.6 *Potassium Iodide Solution* — approximately 10 percent (*w/v*), iodate-free.

4.3.7 *Standard Sodium Thiosulphate Solution* — approximately 0·1 N.

4.3.8 *Starch Solution* — same as in **2.3.4.**

4.4 Procedure — Transfer the ash as obtained in LC : 3 to a platinum crucible (or alternatively, 5 g of the ground leather, accurately weighed, may be used directly in the crucible if an accurate value of the ash is not desired) and mix the ash thoroughly with 5 g of a fusion mixture composed of equal parts of anhydrous sodium carbonate and potassium chlorate. Fuse the contents of the crucible for 30 minutes. Allow the crucible to cool and then transfer it to a beaker containing sufficient hot distilled water to cover it. Digest until the melt no longer adheres to the crucible. Remove the crucible and rinse thoroughly with hot water containing a few drops of concentrated hydrochloric acid. Dissolve the melt by the addition of dilute hydrochloric acid and then gradually add ammonium hydroxide solution till the solution is faintly alkaline. Boil the solution. Filter off precipitated ferric and aluminium hydroxides, and wash the precipitate thoroughly collecting the filtrate and the washings in a 500-ml volumetric flask. Cool solution and then make up the volume up to 500-ml. Transfer exactly 100 ml of the solution to a suitable flask and follow rest of the procedure as given in **2.4.**

4.5 Calculation — same as in **2.5.**

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*Specification for sodium carbonate, anhydrous (*revised*).

†Specification for hydrochloric acid (*revised*).

DETERMINATION OF ALUMINIUM AS ALUMINA (Al_2O_3) IN ALUM TANNED LEATHER

[LC : 11]

Method A

1. SCOPE

1.1 This method prescribes a procedure for the determination of aluminium in leathers, tanned with aluminium salts alone.

2. OUTLINE OF THE METHOD

2.1 In this method, an aluminium tartrate complex is formed. When treated in slightly basic solution with potassium fluoride, this liberates an amount of base equivalent to the aluminium present, which may be titrated.

3. APPARATUS

3.1 Conical Flask — 100 ml capacity.

3.2 Hot-Plate

3.3 Muffle Furnace — provided with pyrometer and thermostatic control.

4. REAGENTS

4.1 Concentrated Hydrochloric Acid — *see* IS : 265-1962*.

4.2 Thymol Blue and Cresol Red Indicator — Prepared by mixing 3 volumes of 0·1 percent alcoholic solution of thymol blue and one volume of 0·1 percent alcoholic solution of cresol red.

4.3 Potassium Hydroxide Solution — approximately 5 N.

4.4 Potassium Tartrate Solution — approximately 50 percent solution neutralized to indicator as given in **4.2**.

4.5 Potassium Fluoride Solution — approximately 20 percent solution neutralized to indicator as given in **4.2**.

4.6 Standard Hydrochloric Acid — approximately 0·1 N.

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*Specification for hydrochloric acid (*revised*).

5. PROCEDURE

5.1 Sample the leather and prepare a test portion for chemical testing in accordance with IS : 5868-1969*. Ash 1 to 2 g of accurately weighed leather from the test portion in a crucible at 800±25°C in a muffle furnace. Likiviate the ash with 5 to 10 ml concentrated hydrochloric acid and then with water. Transfer the contents to a conical flask, and heat on a hot-plate in a fume chamber till the volume is reduced to 5 ml. Add a few drops of the mixed thymol blue cresol red indicator solution and neutralize the excess of acid with potassium hydroxide solution as indicated by disappearance of red colour. Cool the solution and add 20 ml of potassium tartrate solution and complete the neutralization of the acid with potassium hydroxide solution, as indicated by the appearance of pink colour. During the final stages of neutralization of excess acid, use decinormal potassium hydroxide solution. Subsequently add 20 ml of potassium fluoride solution and titrate with standard hydrochloric acid. The end point is reached when the solution assumes the same pink colour which goes over to orange-yellow with further addition of acid.

6. CALCULATION

6.1 Calculate aluminium, as follows:

$$\text{Aluminium (as } Al_2O_3 \text{), percent by weight} = \frac{1{\cdot}7\ VN}{W}$$

where

V = volume in ml of standard hydrochloric acid required for the titration,

N = normality of standard hydrochloric acid, and

W = weight in g of the sample taken for the test.

Method B

(*Adapted from Provisional Method of IUC/16*)

7. SCOPE

7.1 This method describes a procedure for determining the quantity of aluminium in leather. It does not define the chemical identity of the aluminium compounds present in leather and results are expressed in terms of the oxide (Al_2O_3).

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*Methods of sampling for leather.

8. OUTLINE OF THE METHOD

8.1 Leather is ashed to remove organic matter and the inorganic residue brought into solution by alkaline fusion or wet oxidation and the aluminium determined by complexometric titration with disodium cyclohexane-1, 2-diamine tetra-acetate (CDTA) solution.

9. APPARATUS

9.1 Analytical Balance — sensitivity 0·001 g.

9.2 Burette — graduated in 0·1-ml divisions.

9.3 Conical Flask — 300-ml with splash bulb.

9.4 Filter Apparatus

9.5 Glass Beaker — 250 ml.

9.6 Magnetic Stirrer

9.7 *p*H Measuring Apparatus — fitted with glass and calomel electrodes.

9.8 Pipettes

9.9 Platinum Crucible

9.10 Muffle Furnace

9.11 Tall-Form Beaker

9.12 Volumetric Flask — 100 ml.

10. REAGENTS

10.1 Hexamethylene Tetramine — solid.

10.2 Fusion Mixture — potassium carbonate and potassium chlorate in equimolar proportion (7 : 6).

10.3 Hydrochloric Acid, Concentrated — relative density 1·18.

10.4 Oxidizing Mixture — 70 ml of concentrated sulphuric acid and 230 ml of 60 percent perchloric acid.

10.5 Sodium Hydroxide Solution — 40 percent (*w*/*v*).

10.6 Ammonia — equal volumes of ammonia solution of relative density 0·880 and distilled water.

10.7 Disodium Cyclohexane-1, 2-Diamine Tetra-acetate (CDTA) Solution — prepared by suspending 18·2 g of the free acid monohydrate in 200 ml of distilled water, adding 100 ml of M sodium hydroxide and diluting to about one litre (*p*H 6·3). The solution should be standardized before use by titration with 0·05 M zinc chloride.

10.8 Zinc Chloride — 0·05 M standard solution, prepared by dissolving 3·269 g chemically pure zinc powder in hydrochloric acid and heating on a water-bath, cooling and making up to one litre in graduated flask.

10.9 Xylenol Orange (Indicator) — 0·1 percent aqueous solution. This solution is stable for 2 to 3 weeks.

10.10 Chromium Sulphate or Chrome Alum — solid.

11. PROCEDURE

11.1 Sample the leathers and prepare a test portion for chemical testing in accordance with IS : 5868-1969*.

11.2 Weigh about 2 g from the test portion to an accuracy of 1 mg into a previously tared platinum crucible.

11.3 Ash the same in a muffle furnace at a temperature not exceeding 500°C.

11.4 Mix the ash with a 3 to 5 fold quantity of fusion mixture using platinum wire. Heat the crucible in a muffle furnace at 800°C. When the melt has become homogeneous (about 30 min), cool and place in a tall-form beaker containing sufficient hot distilled water to cover the basin; if necessary heat to dissolve the melt. Filter to remove iron and zirconium, wash the filter thoroughly with hot distilled water, collecting the filtrate and washings in a tall-form beaker. Acidify these with hydrochloric acid (**3.3**) to ensure the solution of the aluminium and to bring the *p*H within the range 1 to 3. Boil the acidified solution to remove carbon dioxide, cool and adjust the *p*H if necessary to bring it within the range 1 to 3. Transfer quantitatively to a volumetric flask (100 ml) and make up to volume with distilled water. This is the analytical solution.

11.5 Transfer the ash to a 300-ml conical flask, rinsing out the crucible with oxidizing mixture, add further oxidizing mixture to a total volume of 15 ml. If chromium is not present in the ash, add about 20 mg of chromium sulphate to act as an indicator. Place a splash bulb in the neck of the flask. Transfer to a fume cupboard and heat gently until the mixture boils gently. Continue to heat until the colour changes from green to golden-yellow, heat for a further minute, remove from the source of heat, cool for a further few minutes, and add approximately 100 ml distilled water (*see* Note). The first 2 to 3 ml of water should be added drop-wise with shaking. Add a few earthenware chips or anti-bump granules and boil for 10 to 15 minutes, to expel chlorine. Cool, test the *p*H, and if necessary, add sodium hydroxide to bring it to within the range 1 to 3. Transfer quantitatively to a graduated flask (100 ml) and make up to volume. This is the analytical solution.

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*Methods of sampling for leather.

NOTE — This operation requires great care, and should be carried out in a fume cupboard with the mouth of the flask pointing away from the operator.

11.6 Place an aliquot of analytical solution containing 20 to 30 mg aluminium oxide (Al_2O_3) in a 250-ml beaker mounted on a magnetic stirrer and add 25·0 ml of CDTA. If a precipitate forms, the solution is insufficiently acid. Adjust the *p*H to approximately 5·5 by adding sodium hydroxide. Buffer by adding 5 g of hexamethylene tetramine. If the solution is still acid after adding, neutralize further with dilute ammonia. Dilute to approximately 100 ml, add several drops of xylenol orange and titrate the excess CDTA with 0·05 M zinc chloride solution. Stirring should be continued during the titration and the *p*H maintained at 5·5. The colour change is lemon-yellow to red.

12. CALCULATION

12.1 Calculate aluminium (as Al_2O_3) from the formula:

$$\text{Aluminium (as } Al_2O_3\text{), percent by weight} = \frac{25 - t_0}{M_0} \times \frac{V_1}{V_2} \times 0{\cdot}255$$

where

t_0 = volume of 0·05 M zinc solution consumed in ml,

V_1 = volume of analytical solution in ml,

M_0 = weight in g of leather taken, and

V_2 = volume of aliquot taken for titration in ml.

NOTE — The factor 0·255 takes into account the dilutions used in this method and other constants.

13. ACCURACY

13.1 Repeatability — Duplicate determinations by the same operator should not differ from each other by more than 0·1 percent calculated on the original mass of leather taken.

13.2 Reproducibility — Differences between laboratories should not differ by more than 0·2 percent calculated on the original mass of leather taken.

DETERMINATION OF ZIRCONIUM

[LC : 12]

1. SCOPE

1.1 This method prescribes a procedure for determining the total quantity of zirconium in leather. It is not possible to define the chemical identity of the zirconium compounds present and results are expressed in terms of the oxide, ZrO_2.

2. OUTLINE OF THE METHOD

2.1 Leather is ashed to remove organic matter and the inorganic residue fractionated to remove interfering ions. Zirconium is precipitated as the phosphate. No attempt is made to remove hafnium which is normally present in small quantities.

3. APPARATUS

3.1 Analytical Balance — sensitivity 0·001 g.

3.2 Conical Flasks — 250 ml and 500 ml.

3.3 Desiccator

3.4 Filter Apparatus and Ashless, Acid Resistant Filter Paper

3.5 Muffle Furnace — preferably electric.

3.6 Measuring Cylinders

3.7 Pipettes

3.8 Platinum Basin or Crucible

3.9 Tall-Form Beaker — 500 ml.

4. REAGENTS

4.1 Potassium Bisulphate — solid.

4.2 Sulphuric Acid — relative density 1·83 (*see* IS : 266-1961*).

4.3 Hydrogen Peroxide — 10 percent by volume [3 percent H_2O_2 (*w*/*v*) aqueous solution].

4.4 Barium Chloride — 10 percent (*w*/*v*) aqueous solution.

4.5 Hydrochloric acid — approximately 3 M.

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*Specification for sulphuric acid (*revised*).

4.6 Ammonium Phosphate — 10 percent (*w*/*v*) aqueous solution.

4.7 Ammonium Nitrate — 5 percent (*w*/*v*) aqueous solution.

5. PROCEDURE

5.1 Sample the leather and prepare a test portion for chemical testing in accordance with IS : 5868-1969*. From the test portion, weigh about 2 g to an accuracy of 1 mg into a previously weighed crucible.

5.2 Ash the leather at a temperature not exceeding 500°C in a muffle furnace. When the ashing appears to be complete, cool. The cooled ash should be broken down to a powder with a fine glass rod or platinum wire and mixed with 10 times its weight of potassium bisulphate; add 3 to 5 drops of sulphuric acid. Heat to a dull red over a Bunsen burner, stirring continuously with a platinum wire. When the melt has become homogeneous, cool and place in a 500-ml tall-form beaker containing sufficient distilled water to cover the basin. Carefully add 25 ml of sulphuric acid with gentle shaking of the beaker. Bring to the boil and continue boiling until all the material has been removed from the basin and is in either solution or fine suspension. It may be necessary to break up the melt with a clean glass rod. Filter to remove any insoluble matter which may be retained for the estimation of silica. Wash the filter paper and funnel with successive portions of hot water (about 90°C). Transfer the filtrate and washings to a 500-ml volumetric flask and make up to volume with distilled water. Pipette a 100-ml aliquot into a 500-ml conical flask, dilute with 100 ml of water and add about 15 to 20 ml of sulphuric acid. Add 15 ml of hydrogen peroxide to complex any titanium which may be present. Heat the solution to 50°C and add very slowly and with efficient stirring, 25 ml of ammonium phosphate, stand for at least two hours, then filter through ashless acid resistant filter paper (a Buchner or other vacuum assisted system is desirable), and wash the precipitate with ammonium nitrate at 40°C until the filtrate is free from sulphate ions. Test with barium chloride. Transfer the filter paper and precipitate to a platinum basin previously ignited at 1 000°C, cooled and weighed. Dry in an oven at 100±2°C for about one hour and ignite at 950 to 1 000°C in a muffle furnace for one hour. Cool in a desiccator and weigh. Repeat the ignition until the change is less than 1 mg on successive weighings.

Note — Where zirconium phosphate is present in leather it will not be solubilized by acid fusion.

6. CALCULATION

6.1 Calculate zirconium as zirconium oxide (ZrO_2) as follows:

$$\text{Zirconium (as } ZrO_2 \text{), percent by weight} = 235 \times \frac{W_1}{W_2}$$

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*Methods of sampling for leather.

where

W_1 = weight in g of zirconium pyrophosphate, and

W_2 = weight in g of leather taken.

NOTE — The factor 235 takes into account the dilutions used in this method and other constants.

7. ACCURACY

7.1 Repeatability — Duplicate determinations by the same operator should not differ from each other by more than 0·2 percent calculated on the original mass of leather taken.

7.2 Reproducibility — Results from different laboratories should not differ from each other by more than 0·5 percent calculated on the original mass of leather taken.

DETECTION OF PRESENCE OF FORMALDEHYDE

[LC : 13]

1. SCOPE

1.1 This method is intended for the detection of the presence of formaldehyde in leathers either by Rosaniline hydrochloride or chromotropic acid.

2. REAGENTS

2.1 Dilute Hydrochloric Acid — 36 percent (*w/v*).

2.2 Sodium Sulphite Solution — 10 percent (*w/v*).

2.3 Grosse-Bohle Reagent — Dilute 1 g of Rosaniline hydrochloride in 500 ml of distilled water and add 125 ml of a 10-percent solution of sodium sulphite and 10 ml of dilute hydrochloric acid. Filter and make up to a volume of 1 litre with water.

2.4 Dilute Sulphuric Acid — 72 Percent Solution — Add 150 ml of concentrated acid to 100 ml of water.

2.5 Chromotropic Acid — solid.

3. PROCEDURE

3.1 Method A — Place 0·5 g of leather, cut into small pieces, in a test-tube; add 7·7 ml of a mixture of 10 parts of distilled water and 1 part of 36 percent hydrochloric acid. Heat until boiling starts and then cool to room temperature. Add 1 ml of Grosse-Bohle reagent and allow to stand for 2 hours. If a greyish blue colouration turning to violet is produced, formaldehyde is present.

3.2 Method B — Add a few drops of distillate in a test-tube, 2 ml of dilute sulphuric acid and some solid chromotropic acid. Heat it over a water-bath for 10 minutes at 60°C. Violet colour indicates the presence of formaldehyde.

DETERMINATION OF FREE FORMALDEHYDE

[LC : 14]

1. SCOPE

1.1 This method prescribes a procedure for the determination of free formaldehyde in leathers, tanned with formaldehyde.

2. OUTLINE OF THE METHOD

2.1 Formaldehyde extracted along with water soluble matter is distilled over and estimated iodimetrically.

3. APPARATUS

3.1 Kjeldahl Flask — 500 ml capacity.

4. REAGENTS

4.1 Dilute Sulphuric Acid — approximately 2 N.

4.2 Sodium Bisulphite Solution — 1·2 percent (*w/v*).

4.3 Standard Iodine Solution — approximately 0·1 N.

4.4 Starch Solution — Triturate 5 g of starch and 0·01 g of mercuric iodide with 30 ml of water in a mortar. Pour the resulting paste into one litre of boiling water; boil for three minutes, allow the solution to cool and decant off the clear liquid.

4.5 Sodium Carbonate Solution — 5 percent solution (*w/v*).

5. PROCEDURE

5.1 Take 250-ml extracts of the water solubles as in LC : 6 and 50 ml of dilute sulphuric acid in a Kjeldahl flask and distil the contents for 45 minutes. Collect the distillate into a receiver containing 20 ml of sodium bisulphite solution. Titrate the solution with standard iodine solution using starch as the indicator. Then add 10 to 20 ml of sodium carbonate solution and titrate the contents again with standard iodine solution and record this value in millilitres.

6. CALCULATION

6.1 Calculate the free formaldehyde as follows:

$$\text{Free formaldehyde, percent by weight} = 20 \times 0{\cdot}015 \times VN$$

where

V = volume in ml of standard iodine solution required for the titration, and

N = normality in N of standard iodine solution.

NOTE — The calculation is based on the assumption that the test has been carried out on 10 g of the sample.

DETERMINATION OF COMBINED OIL

[LC : 15]

1. SCOPE

1.1 This method prescribes a procedure for the determination of combined oil in all types of leathers.

2. OUTLINE OF THE METHOD

2.1 The leather from which free fats, oils and other soluble matters have been extracted with a solvent is digested and treated with ether and the combined oils are extracted. The extract is then evaporated and from the residue, the percentage of the combined oil on the weight of leather is calculated.

3. APPARATUS

3.1 Flask — with reflux condenser.

3.2 Oven — set at 102±2°C.

3.3 Separating Funnel

4. REAGENTS

4.1 Alcoholic Potassium Hydroxide Solution — approximately 8 percent (*w/v*).

4.2 Concentrated Hydrochloric Acid — relative density 1·16 (*see* IS : 265-1962*).

4.3 Ether — peroxide-free ether (*see* IS : 336-1964†).

5. PROCEDURE

5.1 Digest for at least an hour the de-fatted material as reserved under **5.1** of LC : 4 in a flask fitted with a reflux condenser containing 50 ml of alcoholic potassium hydroxide solution on a boiling water-bath. Evaporate the alcohol as far as possible and then dilute the residue with 50 ml of water. Then add slowly 15 ml of concentrated hydrochloric acid and again place the flask on a boiling-water-bath for an hour. Cool the flask and transfer its contents completely into a separating funnel. Shake it with ether and allow the layers to separate. Collect the ether layer in a conical flask. Shake the water layer again with ether and collect the ether layer as before in the same conical flask. Continue this extraction with ether until the fatty acids are completely removed. Transfer the combined ether extracts containing the fatty acids to a clean separating funnel and wash with water to remove mineral acids and subsequently dry over anhydrous sodium sulphate to remove dissolved water. Filter into a weighed conical flask and evaporate the ether on the boiling-water-bath. Then dry in the air-oven at 100±2°C for one hour and weigh.

NOTE — The ether extraction of the fatty acids may also be carried out with ease and advantage in a liquid extractor.

6. CALCULATION

6.1 Express the weight of the residue as percentage on the weight of the material taken under **5.1** of LC : 4.

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*Specification for hydrochloric acid (*revised*).

†Specification for ether (*revised*).

DETERMINATION OF WATER SOLUBLE MAGNESIUM SALTS (EPSOM SALT)

[LC : 16]

(*This method supersedes that in* **15** *of IS : 582-1954**)

1. SCOPE

1.1 This method prescribes a procedure for the determination of water soluble magnesium salts like epsom salt in all types of leather.

2. OUTLINE OF THE METHOD

2.1 Water soluble mineral substances are first extracted from a definite amount of leather. Then by complexometric titration magnesium is determined from the water soluble mineral substances.

3. REAGENTS

3.1 Dilute Hydrochloric Acid — 10 percent (*v/v*).

3.2 Ammonium Chloride Solution — 10 percent (*w/v*).

3.3 Ferric Chloride Solution — 10 percent (*w/v*).

3.4 Nitric Acid — concentrated.

3.5 Ammonium Hydroxide Solution — approximately 2 N.

3.6 Ammonium Oxalate Solution — 5 percent (*w/v*).

3.7 Zinc Granules — free from zinc dust.

3.8 Dilute Hydrochloric Acid — approximately 2 N.

3.9 Ether — (*see* IS : 336-1964†).

3.10 Ethyl Alcohol

3.11 Ammonium Chloride — solid.

3.12 Sodium Hydroxide Solution — approximately 2 N.

3.13 Sodium Chloride — solid.

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*Methods of sampling and test for vegetable and chrome tanned leathers.

†Specification for ether (*revised*).

3.14 Diaminoethanetetra-acetic Acid-disodium Salt (DETA) — 0·01 M. Dry DETA at 80°C for several hours. Weigh 3·722 g of DETA and make up to one litre in a measuring flask.

> NOTE — The solution may be kept for a practically unlimited time only in closed polyethylene vessels.

3.15 Buffer Solution — 54 g of ammonium chloride and 350 ml of 25 percent (*v*/*v*) ammonia solution made up to 1 litre with water. This buffer solution has a *p*H of 10·5.

3.16 Methyl Orange Indicator — 0·15 g of water soluble methyl orange in water and diluted to 500 ml.

3.17 Erichrome Black T — Mix well 1 part of Erichrome black T with 300 parts of sodium chloride (solid). Add the indicator in the solid state to the solution.

3.18 Standard Zinc Solution — In a 100-ml beaker, treat 5 g of granules of zinc with 200 ml of 2 N hydrochloric acid. After 5 minutes, decant the acid and wash the zinc repeatedly with water by decantation. Then wash several times with ethyl alcohol and finally with ether. Dry the granules. Weigh accurately 0·163 4 g of these granules and treat with dilute hydrochloric acid. Heat in a water-bath until the zinc dissolves. Transfer the solution to a 250-ml measuring flask, cool and make up to the mark.

4. PROCEDURE

4.1 Standardization of DETA Solution — Pipette out 25 ml of standard zinc solution in a flask, dilute with 50 ml of water, add concentrated ammonium hydroxide solution until the precipitate first formed is redissolved. Add 5 drops of Erichrome black T indicator and titrate with DETA solution to be standardized until the last tint of red colour just disappears to leave a clear blue colour. Run the titration at least in duplicate. The molarity of DETA solution is given by the equation:

$$M_1 = \frac{V_1 M_2}{V_2}$$

where

M_1 = molarity of DETA solution;

V_1 = volume in ml of zinc solution taken for titration;

M_2 = molarity of zinc solution, given by $\frac{X}{0{\cdot}163\,4} \times 0{\cdot}01$, X being the actual amount of zinc granules taken; and

V_2 = volume in ml of DETA solution taken for titration.

4.2 Elimination of Phosphate Ions — Mix the ash in the crucible with 5 ml of dilute hydrochloric acid and dissolve under slight heat. Rinse the solution in a 250-ml glass beaker and dilute with water to approximately 50 ml. Then add 3 ml of ammonium chloride solution, a few drops of concentrated nitric acid and a few drops of ferric chloride solution. Make the solution alkaline with ammonia, and boil. The precipitate that forms should be quite brown in colour. If it is not, then acidify again with hydrochloric acid, add a few drops of ferric chloride solution and make alkaline with ammonia. Repeat this procedure until the formation of a distinctly brown-coloured precipitate occurs. Boil the solution until the smell of ammonia has nearly disappeared. Filter the solution while warm and wash out the beaker with hot water.

4.3 Elimination of Calcium Ions — Use the filtrate after elimination of the phosphate ions in accordance with **4.2**. Treat the solution of the ash or filtrate with 3 ml of ammonium chloride solution, 7·5 ml of ammonia solution and 1·5 ml of ammonium oxalate solution. Heat the solution to boiling, transfer to a 100-ml measuring flask and adjust to volume; mix and filter through a fluted filter. Pipette 50 ml of the filtrate into a 400-ml glass beaker, dilute with 100 ml water, and determine the magnesium in accordance with **4.4**.

4.4 Determination of Water Soluble Magnesium Salts — Transfer the solution into a 300-ml flask. Rinse the crucible several times with a very small quantity of hydrochloric acid and distilled water; neutralize the solution against methyl orange with ammonia or sodium hydroxide solution and boil for 2 to 3 minutes only. Dilute the solution with 150 ml of distilled water, add 20 ml of buffer solution, adjust to 50°C by cooling or heating and add the indicator until the solution becomes clear red. Titrate with 0·01 M DETA solution until the colour changes to pure blue (with no red tint).

NOTE 1 — If the sulphated ash of water solubles obtained in accordance with LC : 7 is used for the determination of magnesium salt content, it is necessary first to eliminate any interfering phosphate or calcium ions (or both together) if only qualitative tests have shown their presence.

NOTE 2 — Use ash-free filter paper throughout.

5. CALCULATION

5.1 Since 1 ml of 0·01 M DETA corresponds to 0·002 465 g of $MgSO_4$, $7H_2O$:

$$\text{Epsom salt, percent by weight} = \frac{0{\cdot}246\,5 \times V}{W}$$

where

V = volume in ml of 0·01 M DETA solution used in the titration, and

W = weight in g of the material taken for the test.

5.2 Permissible Error — The results of a duplicate determination shall not vary by more than ±0·2 percent of epsom salt content in the leather.

DETERMINATION OF GLUCOSE

[LC : 17]

(*This method supersedes that in* **14** *of IS : 582-1954**)

1. SCOPE

1.1 This method is intended for the determination of sugar content in terms of glucose, in all types of leather.

2. OUTLINE OF THE METHOD

2.1 Water soluble matter in a leather is first extracted. The extract is boiled with Fehling solution whereby copper oxide, corresponding to the sugar content is precipitated. It is first necessary to remove tannins with lead acetate, precipitate the excess lead salt and to invert any non-reducing sugars with hydrochloric acid. Sugar content is determined from the weight of the copper oxide by means of Munson and Walker's Table.

3. REAGENTS

3.1 Lead Acetate Solution [Pb ($C_2H_3O_2$)$_2$, $3H_2O$] — saturated.

3.2 Potassium Oxalate — solid.

3.3 Concentrated Hydrochloric Acid — relative density 1·16 (*see* IS : 265-1962†).

3.4 Anhydrous Sodium Carbonate — *see* IS : 296-1965‡.

3.5 Fehling's Solution No. 1 — Dissolve 34·640 g of copper sulphate ($CuSO_4$ $5H_2O$) in water and dilute to 500 ml. Filter through asbestos.

3.6 Fehling's Solution No. 2 — Dissolve 173 g of potassium sodium tartrate and 50 g of sodium hydroxide in water and dilute to 500 ml. Allow the solution to stand for two days and filter through asbestos.

3.7 Rectified Spirit — *see* IS : 323-1959§.

3.8 Ether — peroxide-free (*see* IS : 336-1964||).

4. PROCEDURE

4.1 Transfer exactly 200 ml of the aqueous leather extract as obtained under LC : 6 into a 500-ml flask. Add 25 ml of lead acetate solution, shake

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*Methods of sampling and test for vegetable and chrome tanned leathers.

†Specification for hydrochloric acid (*revised*).

‡Specification for sodium carbonate, anhydrous (*revised*).

§Specification for rectified spirit (*revised*).

||Specification for ether (*revised*).

for 5 to 10 minutes and filter. (During filtration it is advisable to keep the funnels and beakers covered to avoid evaporation). Add excess of potassium oxalate to the filtrate, stir for about 15 minutes and filter. Repeat the filtration until a clear solution is obtained. Pipette 150 ml of the clear filtrate into a 600-ml conical flask. Add 5 ml of concentrated hydrochloric acid and boil under reflux condenser for 2 hours to convert cane sugar into reducing sugars. Cool the solution, neutralize with anhydrous sodium carbonate (use litmus paper), and transfer completely to a 200-ml volumetric flask. Make up the volume to 200 ml and filter through a double filter paper till clear. Determine the glucose in the solution immediately as in **4.2**.

4.2 Pipette 25 ml of Fehling's solution No. 1 and also 25 ml of the Fehling's solution No. 2 into a 400-ml beaker. Add to the contents of the beaker, 50 ml of the neutralized and clarified solution (this 50 ml corresponds to one-sixth of the original 200 ml of leather extract) obtained under **4.1** and bring this solution to boiling in exactly 4 minutes. Then boil further for 2 minutes. Filter immediately through asbestos (*see* Note) in a Gooch crucible, wash thoroughly with hot water, then with rectified spirit and finally with ether. Dry the washed residue for half an hour in a steam-bath and weigh as cuprous oxide. The 50-ml aliquot taken for the determination of glucose corresponds to one-fifteenth of the total water extract (500 ml).

NOTE 1 — The finely divided long fibred asbestos should be digested with dilute nitric acid (1 part of concentrated nitric acid to 3 parts of water) for 2 to 3 days, washed free from acid and then digested for the same period with 10 percent (*w/v*) of sodium hydroxide solution and finally washed free from alkali. In preparing the Gooch crucible, make a bottom layer of about 6 mm of the coarser fibres and top off with a layer of fine asbestos. Wash the mat so prepared with boiling Fehling's solution (the mixture of the copper sulphate and alkaline tartrate solution), then with nitric acid (1 : 3) and finally with hot water. Gooch crucibles so prepared with asbestos mats may be used repeatedly in successive determinations.

NOTE 2 — The rate of heating by the flame should be regulated by a screw clip before the sugar determinations are commenced. This is best done by adjusting the flow of gas so as to bring 25 ml of copper solution plus 25 ml of alkaline tartrate solution plus 50 ml of water in a 400-ml beaker to boiling in exactly 4 min.

NOTE 3 — Owing to their appreciable solubility in the alkaline reagents sintered glass filter crucibles should not be used. Sintered porcelain crucibles have been found to be satisfactory.

NOTE 4 — If the aqueous extract gives a negative test for tannin within lead acetate it may be hydrolyzed without the preliminary detannization. If this shortened porcedure is used some adjustment in the calculations will be required.

5. CALCULATION

5.1 Determine the percentage of sugar in terms of glucose by the use of the Munson and Walker's Table (Table 1).

TABLE 1 MUNSON AND WALKER'S TABLE — RELATION OF CUPROUS OXIDE AND GLUCOSE

(*Clause* 5.1)

(Expressed in milligrams)

CUPROUS OXIDE (Cu_2O) (1)	GLUCOSE (2)	CUPROUS OXIDE (Cu_2O) (1)	GLUCOSE (2)	CUPROUS OXIDE (Cu_2O) (1)	GLUCOSE (2)	CUPROUS OXIDE (Cu_2O) (1)	GLUCOSE (2)
10	4·0	50	21·3	90	38·9	130	56·8
11	4·5	51	21·7	91	39·3	131	57·2
12	4·9	52	22·2	92	39·8	132	57·7
13	5·3	53	22·6	93	40·2	133	58·1
14	5·7	54	23·0	94	40·6	134	58·6
15	6·2	55	23·5	95	41·1	135	59·0
16	6·6	56	23·9	96	41·5	136	59·5
17	7·0	57	24·3	97	42·0	137	60·0
18	7·5	58	24·8	98	42·4	138	60·4
19	7·9	59	25·2	99	42·9	139	60·9
20	8·3	60	25·6	100	43·3	140	61·3
21	8·7	61	26·1	101	43·8	141	61·8
22	9·2	62	26·5	102	44·2	142	62·2
23	9·6	63	27·0	103	44·7	143	62·7
24	10·0	64	27·4	104	45·1	144	63·1
25	10·5	65	27·8	105	45·5	145	63·6
26	10·9	66	28·3	106	46·0	146	64·0
27	11·3	67	28·7	107	46·4	147	64·5
28	11·8	68	29·2	108	46·9	148	65·0
29	12·2	69	29·6	109	47·3	149	65·4
30	12·6	70	30·0	110	47·8	150	65·9
31	13·1	71	30·5	111	48·2	151	66·3
32	13·5	72	30·9	112	48·7	152	66·8
33	13·9	73	31·4	113	49·1	153	67·2
34	14·3	74	31·8	114	49·6	154	67·7
35	14·8	75	32·2	115	50·0	155	68·2
36	15·2	76	32·7	116	50·5	156	68·6
37	15·6	77	33·1	117	50·9	157	69·1
38	16·1	78	33·6	118	51·4	158	69·5
39	16·5	79	34·0	119	51·8	159	70·0
40	16·9	80	34·4	120	52·3	160	70·4
41	17·4	81	04·9	121	52·7	161	70·9
42	17·8	82	35·3	122	53·2	162	71·4
43	18·2	83	35·8	123	53·6	163	71·8
44	18·7	84	36·2	124	54·1	164	72·3
45	19·1	85	36·7	125	54·5	165	72·8
46	19·6	86	37·1	126	55·0	166	73·2
47	20·0	87	37·5	127	55·4	167	73·7
48	20·4	88	38·0	128	55·9	168	74·1
49	20·9	89	38·4	129	56·3	169	74·6

(*Continued*)

TABLE 1 MUNSON AND WALKER'S TABLE — RELATION OF CUPROUS OXIDE AND GLUCOSE — *Contd*

CUPROUS OXIDE (Cu_2O)	GLUCOSE	CUPROUS OXIDE (Cu_2O)	GLUCOSE	CUPROUS OXIDE (Cu_2O)	GLUCOSE	CUPROUS OXIDE (Cu_2O)	GLUCOSE
(1)	(2)	(1)	(2)	(1)	(2)	(1)	(2)
170	75·1	210	93·7	250	112·8	290	132·3
171	75·5	211	94·2	251	113·2	291	132·7
172	76·0	212	94·6	252	113·7	292	133·2
173	76·4	213	95·1	253	114·2	293	133·7
174	76·9	214	95·6	254	114·7	294	134·2
175	77·4	215	96·1	255	115·2	295	134·7
176	77·8	216	96·5	256	115·7	296	135·2
177	78·3	217	97·0	257	116·1	297	135·7
178	78·8	218	97·5	258	116·6	298	136·2
179	79·2	219	98·0	259	117·1	299	136·7
180	79·7	220	98·4	260	117·6	300	137·2
181	80·1	221	98·9	261	118·1	301	137·7
182	80·6	222	99·4	262	118·6	302	138·2
183	81·1	223	99·9	263	119·0	303	138·7
184	81·5	224	100·3	264	119·5	304	139·2
185	82·0	225	100·8	265	120·0	305	139·7
186	82·5	226	101·3	266	120·5	306	140·2
187	82·9	227	101·8	267	121·0	307	140·7
188	83·4	228	102·2	268	121·5	308	141·2
189	83·9	229	102·7	269	122·0	309	141·7
190	84·3	230	103·2	270	122·5	310	142·2
191	84·8	231	103·7	271	122·9	311	142·7
192	85·3	232	104·1	272	123·4	312	143·2
193	85·7	233	104·6	273	123·9	313	143·7
194	86·2	234	105·1	274	124·4	314	144·2
195	86·7	235	105·6	275	124·9	315	144·7
196	87·1	236	106·0	276	125·4	316	145·2
197	87·6	237	106·5	277	125·9	317	145·7
198	88·1	238	107·0	278	126·4	318	146·2
199	88·5	239	107·5	279	126·9	319	146·7
200	89·0	240	108·0	280	127·3	320	147·2
201	89·5	241	108·4	281	127·8	321	147·7
202	89·9	242	108·9	282	128·3	322	148·2
203	90·4	243	109·4	283	128·8	323	148·7
204	90·9	244	109·9	284	129·3	324	149·2
205	91·4	245	110·4	285	129·8	325	149·7
206	91·8	246	110·8	286	130·3	326	150·2
207	92·3	247	111·3	287	130·8	327	150·7
208	92·8	248	111·8	288	131·3	328	151·2
209	93·2	249	112·3	289	131·8	329	151·7

TABLE 1 MUNSON AND WALKER'S TABLE — RELATION OF CUPROUS OXIDE AND GLUCOSE — *Contd*

Cuprous Oxide (Cu_2O) (1)	Glucose (2)	Cuprous Oxide (Cu_2O) (1)	Glucose (2)	Cuprous Oxide (Cu_2O) (1)	Glucose (2)	Cuprous Oxide (Cu_2O) (1)	Glucose (2)
330	152·2	370	172·7	410	193·7	450	215·2
331	152·7	371	173·2	411	194·2	451	215·8
332	153·2	372	173·7	412	194·7	452	216·3
333	153·7	373	174·2	413	195·2	453	216·9
334	154·2	374	174·7	414	195·8	454	217·4
335	154·7	375	175·3	415	196·3	455	218·0
336	155·2	376	175·8	416	196·8	456	218·5
337	155·8	377	176·3	417	197·4	457	219·1
338	156·3	378	176·8	418	197·9	458	219·6
339	156·8	379	177·3	419	198·4	459	220·2
340	157·3	380	177·9	420	199·0	460	220·7
341	157·8	381	178·4	421	199·5	461	221·3
342	158·3	382	178·9	422	200·1	462	221·8
343	158·8	383	179·4	423	200·6	463	222·4
344	159·3	384	180·0	424	201·1	464	222·9
345	159·8	385	180·5	425	201·7	465	223·5
346	160·3	386	181·0	426	202·2	466	224·0
347	160·8	387	181·5	427	202·8	467	224·6
348	161·4	388	182·0	428	203·3	468	225·1
349	161·9	389	182·6	429	203·8	469	225·7
350	162·4	390	183·1	430	204·4	470	226·2
351	162·9	391	183·6	431	204·9	471	226·8
352	163·4	392	184·1	432	205·5	472	227·4
353	163·9	393	184·7	433	206·0	473	227·9
354	164·4	394	185·2	434	206·5	474	228·5
355	164·9	395	185·7	435	207·1	475	229·0
356	165·4	396	186·2	436	207·6	476	229·6
357	166·0	397	186·8	437	208·2	477	230·1
358	166·5	398	187·3	438	208·7	478	230·7
359	167·0	399	187·8	439	209·2	479	231·3
360	167·5	400	188·4	440	209·8	480	231·8
361	168·0	401	188·9	441	210·3	481	232·4
362	168·5	402	189·4	442	210·9	482	232·9
363	169·0	403	189·9	443	211·4	483	233·5
364	169·6	404	190·5	444	212·0	484	234·1
365	170·1	405	191·0	445	212·5	485	234·6
366	170·6	406	191·5	446	213·1	486	235·2
367	171·1	407	192·1	447	213·6	487	235·7
368	171·6	408	192·6	448	214·1	488	236·3
369	172·1	409	193·1	449	214·7	489	236·9
						490	237·4

DETERMINATION OF pH AND DIFFERENCE FIGURE OF WATER SOLUBLE MATTER

[LC : 18]

(*This method supersedes that in* **13** *of IS : 582-1954**)

1. SCOPE

1.1 This method prescribes a procedure for the determination of pH and Difference Figure for all types of leather in all stages of dressing. pH value and difference figure relate to the leather extract got with a 20-fold quantity of distilled water.

2. PRINCIPLE OF THE METHOD

2.1 Leather always contains certain quantities of acid (less often of alkali), which determines its pH. Free strong acid or alkali may damage the leather in course of time, and possibly other material treated with it. The pH value of the aqueous leather extract and its Difference Figure serve to measure the acidity or alkalinity of the leather. Both depend on the quantity and strength of the acid or base contained in the leather and on the presence of buffer salts. If the pH value of the extract is below 4 or over 10, the Difference Figure is:

a) large in the presence of strong acid or base and absence of buffering salts; and

b) small in the presence of strong acid or base and considerable quantities of buffering salts or in the presence of weak acid or base (independent of the buffering salt present).

2.2 In the preparation of the aqueous extract, equilibrium is attained more or less quickly between the acid or base going into solution and that remaining in the sample material, according to the type of leather and the degree to which it is ground. The extraction of leather which is difficult to wet may cause difficulty in pH determination.

2.3 The pH value and Difference Figure of the aqueous extract are taken into consideration when deciding whether a leather contains dangerous amounts of free strong acid or base, which is why, in border-line cases, both factors should be determined with adequate accuracy.

2.4 The method described aims at the reproducible and sufficiently accurate determination of the pH value of an aqueous leather extract and its Difference Figures.

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*Methods of sampling and test for vegetable and chrome tanned leathers.

3. APPARATUS

3.1 Flask — 200 ml, wide-necked, polyethylene, or similar flask with ground glass stopper, consisting of resistant glass of minimum alkalinity.

3.2 *p*H Meter

3.2.1 *Measuring System* — range 0 to 14 *p*H, reading to an accuracy of 0·05 *p*H (scale division 0·1 *p*H) or apparatus with higher accuracy.

3.2.2 *Electrode System* — glass electrode with separate or built-in reference electrode (combined glass electrode), calibrated with buffer solution of known *p*H value.

3.2.2.1 Glass electrodes with spherical or cylindrical membranes are suitable. Glass electrodes are recommended which are of universal application in the entire *p*H range of 0 to 14, the alkali errors of which are first taken into consideration at a *p*H value exceeding 2·5.

3.3 Shaker Apparatus — shaker apparatus set at a speed of 50±10 rev/min.

4. REAGENTS

4.1 Buffer Solution — The buffer solutions as given in **4.1.1** and **4.1.2** for calibration of the electrode system are recommended.

NOTE — Buffer errors transfer themselves to measurement results. It is preferable to use freshly made buffer solutions. Used buffer solutions should not be put back into the stock solutions; either pour it away or keep it in a small flask to be used again.

4.1.1 *Standard I* — 0·05 M potassium hydrogen phthalate, *p*H 4·00±0·02 at 27°C.

4.1.2 *Standard II* — 0·01 M dibasic sodium tetraborate, *p*H 9·22±0·02 at 27°C.

4.2 Distilled Water — *p*H value between 6·0 and 7·0 and specific conductivity not greater than 2×10^{-6} ohm^{-1} cm^{-1} at 27°C. If it is desired to stock distilled water, a 10-litre aspirator bottle of polyethylene, with nylon or PVC tap and a polyethylene stopper carrying a soda lime tube is recommended. The *p*H of the water should be controlled.

NOTE — Water distilled with ion exchangers may be used if it fulfils the purity requirements given above.

5. PROCEDURE

5.1 Sample the leather and prepare a test portion for chemical testing in accordance with IS : 5868-1969*. Weigh 5·0±0·1 g from the test portion and take in a polyethylene flask bottle; shake well with 100 ml of water for 30 seconds to wet the ground leather uniformly. Then shake mechanically in the shaker for 16 to 24 h. Determine the *p*H value of the unfiltered extract.

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*Methods of sampling for leather.

5.2 To determine the Difference Figure, dilute 10 ml of the decanted extract to 100 ml with water. Rinse the electrodes with approximately 20 ml of this diluted solution and then measure the *p*H. Read off the *p*H value of the extract and the diluted extract solution to an accuracy of 0·01 to 0·02 *p*H after constant value has been attained on the measuring system.

NOTE 1 — If the *p*H value is between 4 and 10, Difference Figure need be measured.

NOTE 2 — For *p*H measurements by glass electrode, a calibration of the measuring electrode by solutions of known *p*H value (buffer solution) is necessary. As a rule, calibration with one buffer solution is sufficient. The glass electrode should be checked from time to time by measurement in two different buffers.

NOTE 3 — As the accuracy of *p*H measurement is higher, the smaller the *p*H differences between measurement value and calibrated point, calibration value as far as possible, should not be more than ± 2 *p*H from the measuring value.

NOTE 4 — Aqueous extracts of heavily fatliquored leather may, in time, make the electrode membrane rather dirty. In such case it is advantageous to rub the membrane lightly with a piece of cottonwool dipped in acetone or to hang the electrode in a 1 : 1 water-acetone mixture. After cleaning with acetone the electrode membrane should again be thoroughly soaked in water.

NOTE 5 — All apparatus should be of low soluble content and be sufficiently insoluble in water for the requirements of *p*H measurement. It is advisable to test the apparatus carefully before use by a blank experiment using distilled water. Test the *p*H value and the conductivity before and after the experiment.

NOTE 6 — The test solution may be used for qualitative tests for chlorides and sulphates and conductivity measurements.

5.3 *p*H value and Difference Figure of damp leather may also be determined in the factory. The material for this should not be ground but it should be subdivided in a suitable way by stamping or cutting. The water content of the damp leather should be estimated, and a larger quantity of leather should be taken for making the aqueous extract.

6. REPORT

6.1 The report shall give the following:

a) A reference of this method;

b) Details of all deviation from the prescribed test condition;

c) A reference to any instability of the *p*H or Difference Figure; and

d) Statement of the mean value of the individual determination of *p*H value and if this is below 4 or over 10, the Difference Figure exact to 0·05 *p*H units.

DETERMINATION OF TOTAL CHLORIDES

[LC : 19]

1. SCOPE

1.1 This method is intended for the determination of total chlorides (as sodium chloride), present in all types of leather.

2. OUTLINE OF THE METHOD

2.1 A known quantity of the leather is ashed at a controlled temperature and the chloride content in the aqueous extract of the same is determined volumetrically.

3. REAGENTS

3.1 Concentrated Nitric Acid — *see* IS : 264-1968*.

3.2 Standard Silver Nitrate Solution — 0·05 N.

3.3 Calcium Carbonate — solid.

3.4 Potassium Chromate Solution — 5 percent (*w*/*v*).

4. PROCEDURE

4.1 Preparation of Aqueous Extract — Ash about 10 g of accurately weighed ground leather and ignite in a muffle furnace at 500 to 550°C. Moisten the ash with six drops of concentrated nitric acid in water, and make up to 500 ml in a volumetric flask.

4.2 Pipette out 100 ml of the above solution in a flask, neutralize the acidity by the addition of calcium carbonate, add 1 ml of potassium chromate solution and titrate against silver nitrate solution, until a red tinge precipitate or solution is obtained.

5. CALCULATION

5.1 Calculate the amount of chlorides in the material as follows:

$$\text{Chlorides (as NaCl), percent by weight} = \frac{29{\cdot}23 \times VN}{W}$$

where

V = volume in ml of the standard silver nitrate solution,

N = normality in N of standard silver nitrate solution, and

W = weight in g of the material taken for ashing.

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*Specification for nitric acid (*first revision*).

DETERMINATION OF TOTAL SULPHATES

[LC : 20]

1. SCOPE

1.1 This method is intended for the determination of total sulphates (as sodium sulphate), present in all types of leather.

2. OUTLINE OF THE METHOD

2.1 A known quantity of the leather is ashed at a controlled temperature and total sulphates in the aqueous extract determined gravimetrically.

3. REAGENTS

3.1 Concentrated Hydrochloric Acid — relative density 1·16 (*see* IS : 265-1962*).

3.2 Barium Chloride Solution — Dissolve about 12 g of barium chloride crystals in water and make up the volume to 100 ml.

4. PROCEDURE

4.1 Take 100 ml of extract, prepared as in **4.1** of LC : 19, in a 600-ml beaker, and add to it 10 ml of concentrated hydrochloric acid. Filter the solution and dilute the filtrate to about 350 ml. Heat to boiling and add 10 ml of hot barium chloride solution. Keep the reaction mixture on a water-bath for half an hour and then let it stand overnight. Filter the precipitate through a Gooch crucible. Wash the precipitate free from chlorides, dry it and then ignite to constant weight. Carry out a blank test using the same quantities of reagents as used for the experiment with the material.

5. CALCULATION

5.1 Calculate the amount of sulphates in the material as follows:

$$\text{Sulphates (as } Na_2SO_4 \text{), percent by weight} = 304{\cdot}7 \times \frac{W_1}{W_2}$$

where

W_1 = weight in g of the residue obtained, and

W_2 = weight in g of the material taken for ashing.

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*Specification for hydrochloric acid (*revised*).

DETERMINATION OF BOUND ORGANIC SUBSTANCES AND DEGREE OF TANNAGE

[LC : 21]

1. SCOPE

1.1 This method prescribes a procedure for the determination of bound organic substances and degree of tannage of vegetable tanned leather.

2. OUTLINE OF THE METHOD

2.1 In this method, percentage of volatile matter, total ash, extractable substances, hide substances and water soluble organic substances in a leather are first determined. Then the bound organic substances and degree of tannage is calculated.

3. PROCEDURE

3.1 Determine the volatile matter, percent by weight, in accordance with the method given in LC : 1.

3.2 Determine total ash, percent by weight, in accordance with the method given in LC : 3.

3.3 Determine solvent extractable substances, percent by weight, in accordance with the method given in LC : 4.

3.4 Determine hide substances, percent by weight, in accordance with the method given in LC : 5.

3.5 Determine water soluble organic substances, percent by weight, in accordance with the method given in LC : 9.

4. CALCULATION

4.1 Calculate bound organic substances as follows:

$$\text{Bound organic substances, percent by weight} = 100 - X$$

where

X = the value obtained by summing up all the values obtained in **3.1** to **3.5**.

NOTE — In case of combination tanned leathers, a factor may be used to estimate the content of chrome tanning substances.

4.1.1 Bound organic substance, percent by weight, may also be calculated as the difference between 100 and the sum of the percentages by weight of volatile matter (LC : 1), solvent extractable substances (LC : 4) water soluble matter (LC : 6), water insoluble ash (LC : 8) and hide substance (LC : 5).

4.2 Calculate degree of tannage as follows:

$$\text{Degree of tannage} = 100 \times \frac{A}{B}$$

where

A = bound organic substances, percent by weight (*see* **4.1** and **4.1.1**); and

B = hide substance, percent by weight (*see* **3.4**).

TEST FOR CORROSION PRODUCED BY LEATHER IN CONTACT WITH METAL

[LC : 22]

(*Adapted from ASTM Designation: D-1611-60*)

1. SCOPE

1.1 This method prescribes qualitative and quantitative test procedures to determine corrosion produced by leather in contact with metal.

2. TERMINOLOGY

2.1 Corrosion — A modification of the metal surface in contact with a leather test piece, evidenced by visible pitting and erosion of the metal surface or by a change in weight of the metal tested in comparison with a blank determination. Any permanent discoloration in film form on the test piece which does not readily buff off with a non-abrasive cloth shall be called incipient corrosion. In the latter case and for quantitative purposes, the corrosion level should be assessed by determination of the weight changes which occur in the metal panel.

3. PRINCIPLE OF THE METHOD

3.1 Significance of the Method — The procedure is primarily intended to evaluate chrome leather intended for use in hydraulic systems under circumstances where metal corrosion is a serious factor. A high relative humidity is used for the purpose of accelerating the results.

3.2 Outline of the Method — A conditioned piece of leather and two plates of the same metal are dipped in a non-corrosive oil and drained to the drip point. They are stacked together under a load of 10 ± 1 kg and placed in a desiccator containing a relative humidity of 90 to 95 percent at 27 ± 1°C for a period of 14 days. For quantitative purposes, the metal plates are weighed before and after the test.

4. APPARATUS

4.1 Analytical Balance

4.2 Desiccator — containing a saturated solution of ammonium dihydrogen phosphate.

4.3 Forceps

4.4 Metal Plates — four metal plates whose surface roughness should be 0·16 to 0·32 microns R_a value, conforming to IS : 3073-1967*. They should be approximatelp 50 × 100 mm rectangular pieces and uniform in thickness.

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*Assessment of surface roughness.

4.5 Non-abrasive Cloth — The cloth should not scratch or mar the surface of the metal plates when the plates are rubbed with the cloth, or cause any change in the weight of the metal plates.

4.6 Piece of Glass — approximately of the length and width of the metal panels.

4.7 Weight — 10±1 kg.

5. REAGENTS

5.1 Non-corrosive Oil — white oil conforming to IS : 1083-1957*.

5.2 Petroleum Hydrocarbon Solvent — solvent 50/120 conforming to IS : 1745-1966†.

6. TEST PIECE

6.1 The test piece shall be a piece of leather, 25×80 mm and split to a uniform thickness as specified in the order or contract.

7. PROCEDURE

7.1 Qualitative Test (Method A) — Condition the leather test piece by placing it in a desiccator at 90 to 95 percent relative humidity and 27±1°C for a period of 72 hours. Buff the metal plates with a non-abrasive cloth and wash them with the solvent specified in **5.2**. Immerse the conditioned test piece and the metal plates in a non-corrosive oil and drain them to the drip point. Place the test piece between the two metal plates, hold them together under a load of 10±1 kg, and place them in the desiccator. Insert a glass plate between the top metal plate and the weight to avoid contact between dissimilar metals. Also prepare two control plates in the same manner. Place them in the desiccator so that they do not touch each other or the test piece. Then close the desiccator and hold the test piece and plates at 90 to 95 percent relative humidity at 27±1°C for a period of 14 days. At the end of 14 days, inspect the surfaces of the plates which were in contact with the leather test pieces and compare them with the control plates. To facilitate inspection and evaluation, the plates may be washed with solvent specified in **5.2** and buffed with the non-abrasive cloth.

7.2 Quantitative Test (Method B) — For quantitative evaluation, the procedure shall be the same as in **7.1**, except that the metal plates shall be handled with forceps and weighed to the nearest milligram after buffing and washing with solvent specified in **5.2** and before dipping in the non-corrosive oil. At the conclusion of the test, weigh the plates again after washing in solvent specified in **5.2** and buffing with the non-abrasive cloth. Handle the two metal control plates having the same dimensions as the test plates in exactly the same manner as the test plates with the exception that they shall not be brought into contact with the test pieces. At the conclusion of the test, apply and change in weight of the control plates as a correction to the change in weight of the test plates. The result is the change in weight of the test plates from being in contact with the leather test piece.

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*Specification for white oil, light, technical.

†Specification for petroleum hydrocarbon solvents (*first revision*).

8. REPORT

8.1 Qualitative Test (Method A) — For qualitative purposes, report the leather as being either non-corrosive, causing staining, causing incipient corrosion, or causing corrosion.

8.2 Quantitative Test (Method B) — For quantitative purposes, if visible pitting is evident, record the total number of pit marks. If erosion is evident, report the percent area of erosion. In the absence of visible pitting or erosion, express the corrosion level as a gain or loss in milligrams per square centimetre. This is obtained by dividing the change in weight of the test plates from being in contact with the test piece (*see* **7.2**) by the area of the leather test piece in contact with the plates. If there is no change in the weight of the plates from being in contact with the test piece, report the leather as being non-corrosive.

9. ACCURACY

9.1 The accuracy of the quantitative results depends on the accuracy of the measurement of the weight changes of the metal panels, the method of determining the area of erosion, and correctly counting pit marks.

DETERMINATION OF SILICONE

[LC : 23]

(*Adapted from Provisional Method of IUC 14*)

Method A

1. SCOPE

1.1 This method prescribes a procedure for determining the total quantity of silicone in leather.

2. OUTLINE OF THE METHOD

2.1 Leather is oxidized in a Parr Bomb under alkaline conditions to destroy organic matter and to solubilize the mineral matter. The silicone is determined absorptiometrically as a silico-molybdate complex.

2.2 It is not possible to define the chemical identity of the silicone compounds present in leather and results are expressed in terms of the oxide (SiO_2). Organo-silanes may be present in leather and they may be determined by this procedure. Organo-silanes are almost quantitatively extracted from leather by tetrahydrofuran or dichloromethane, and this forms the basis of a method for their determination. A more reliable estimate of inorganic silicone-containing compounds will, therefore, be obtained if leathers suspected of containing organo-silanes are first extracted with dichloromethane.

3. APPARATUS

3.1 Analytical Balance — sensitivity 0·001 g.

3.2 Beaker — 250 ml.

3.3 Graduated Flasks — 100 and 500 ml.

3.4 Parr Bomb — 22 ml.

3.5 *p*H Meter with Glass and Reference Electrodes

3.6 Pipettes

3.7 Spectrophotometer or Photo-Electric Absorptiometer

4. REAGENTS

4.1 Concentrated Sulphuric Acid — relative density 1·83, diluted 1 : 2 (*v*/*v*) with water (*see* IS : 266-1961*).

4.2 Standard Sulphuric Acid — 1·0 N.

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*Specification for sulphuric acid (*revised*).

4.3 Glycerol

4.4 Sodium Sulphate — 10 percent (*w/v*), aqueous solution.

4.5 Ammonium Molybdate — 10 percent (*w/v*), aqueous solution, containing 2 ml of ammonia (relative density 0·880) per 100 ml of solution.

4.6 Stannous Chloride — 1 percent solution prepared as follows:

Dissolve 10 g of stannous chloride ($SnCl_2 2H_2O$), dissolved in 12 ml of warm hydrochloric acid (relative density 1·19) and 5 ml of water then diluted to 1 000 ml. This solution should be used freshly prepared.

4.7 Fusion Mixture — consisting of 100 g of sodium peroxide, 10 g of potassium nitrate, and 3 g of powdered sucrose.

5. PROCEDURE

5.1 Sample the leathers and prepare a test portion for chemical testing of leather in accordance with IS : 5868-1969*.

5.2 Fusion — Place 5 g of fusion mixture in the Parr Bomb and followed by approximately 0·5 g weighed to an accuracy of 1 mg from the test portion. Moisten the leather with glycerol approximately 0·5 ml and cover with 20 g of oxidizing mixture. Close the bomb, place in its protection tube and warm its base with a small gas flame for 20 minutes. Allow to cool. Open the bomb and place it in the beaker, rinse the cover with distilled water, collecting the rinsings in the beaker, add sufficient water to dissolve the contents of the bomb, remove it and rinse into the beaker. Transfer the beaker to a steam-bath and leave for two hours or until the hydrogen peroxide is decomposed. Cool, neutralize with sulphuric acid, adding a very slight excess, dilute to 500 ml in a graduated flask to give the analytical solution. Dilute a known volume of analytical solution containing 0·05 to 0·20 mg silicone dioxide (usually 10 ml) with 40 ml of water and acidify to *p*H 1·8 with sulphuric acid. Transfer quantitatively to a 100-ml graduated flask. Add 5 ml of sodium sulphate solution and 0·8 ml of ammonium molybdate solution and stand for 30 minutes. Add 10 ml of sulphuric acid and 10 ml of stannous chloride, make up to 100 ml with water. Stand for 30 minutes. Measure the blue coloration on the absorptiometer or spectrophotometer at $\lambda=630$ nm. The colour is stable up to 1 hour.

5.3 A standard curve may be prepared by fusing 0·1 g of pure silica, weighed to the nearest 1 mg, with sodium carbonates and making up to volume; the colour is developed in aliquots of this solution by the method described above the treatment of the analytical solution.

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*Methods of sampling for leather.

NOTE 1 — The acid insoluble residue obtained in the isolation of zirconium for analysis by LC : 12 may be substituted for leather at the fusion stage. If followed, this fact should be stated.

NOTE 2 — Alternatively, a Wurzschmitt Bomb may be used in lieu of the Parr Bomb in which case the sample size should be reduced to 100 mg and other quantities reduced accordingly up to and including the preparation of the analytical solution.

6. CALCULATION

6.1 Calculate the silica content in leather from the formula:

$$\text{Silicone (as } SiO_2 \text{) percent by weight} = \frac{50\,000 \times W_1}{V\,W_2}$$

where

W_1 = weight in g of SiO_2 in the aliquot of analytical solution taken for colour development, and

W_2 = weight in g of the sample.

NOTE — The factor 50 000 takes into account the dilutions used in this method.

7. ACCURACY

7.1 Repeatability — Duplicate determinations by the same operator should not differ from each other by more than 0·2 percent calculated on the original weight of leather taken.

7.2 Reproducibility — Differences between laboratories should not differ by more than 0·5 percent calculated on the original weight of leather taken.

Method B

8. SCOPE

8.1 This method describes a procedure for determining the quantity of silicone in all leathers containing silicone.

9. OUTLINE OF THE METHOD

9.1 The silicone determination is made on the tetrahydrofuran (THF) extract of leather by treatment with hydrofluoric acid in the presence of *n*-propanol. The silicone is converted to the corresponding fluorosilane which is lost by evaporation. The quantity of silicone in the extract (and hence in the leather) is determined as the difference in weight of the extract before and after the treatment with hydrofluoric acid and *n*-propanol.

10. APPARATUS

10.1 Analytical Balance — weighing accurately to a milligram.

10.2 Extraction Flask — flat-bottom, 250 ml.

10.3 Measuring Cylinder — polyethylene, 10 ml.

10.4 Oven — set at 100±2°C.

10.5 Platinum Basin — 50 ml capacity.

10.6 Pasteur Pipette

10.7 Soxhlet Condenser — double surface.

10.8 Soxhlet Extractor — 100 ml capacity.

10.9 Soxhlet Thimble — 30 × 100 mm.

11. REAGENTS

11.1 Tetrahydrofuran (THF) — boiling range 65 to 67°C.

11.2 Hydrofluoric Acid — 40 percent.

11.3 *n*-propanol — boiling range 96 to 99°C.

11.4 Hydrocarbon Solvents — boiling range 40 to 60°C.

12. PROCEDURE

12.1 Sample the leathers and prepare a test portion in accordance with IS : 5868-1969*. Weigh accurately 2 to 5 g of ground leather from the test portion into a Soxhlet thimble. Cover the leather with a small quantity of previously degreased cottonwool. Extract the leather in a Soxhlet apparatus for two hours (*see* Note 1).

12.2 Remove the greater part of the solvent from the extract by distillation and transfer the residue (grease+silicone) to a previously dried and weighed platinum basin by means of a Pasteur pipette. Rinse the flask with three 10-ml portions of THF transferring the successive washings to the platinum basin by the aid of the same pipette. Finally, rinse the pipette with a little more solvent collecting the washings in the basin. Remove the solvent by heating the basin on a water-bath and displace the solvent vapour continuously by directing a current of air over the surface of the basin throughout the period of evaporation (*see* Note 2).

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*Methods of sampling for leather.

12.3 Remove the residual traces of THF by three successive additions and evaporations of 10-ml portions of petroleum spirit under the conditions given in **5.2**.

12.4 Dry the basin containing the residue of silicone and grease in the oven at 100±2°C for four hours before weighing. After weighing, return the basin to the oven and weigh at hourly intervals up to seven hours total drying time or until successive weighings do not differ by more than 2 percent of the weight of residue found after four-hours drying.

12.5 The final weight at this stage shall be called weight A.

12.6 Add to the contents of the basin 40 ml *n*-propanol and 2·5 ml of 40 percent aqueous hydrofluoric acid (*see* Note 3). Place the basin on a water-bath and evaporate the contents of the basin almost to dryness (*see* Note 3). Repeat the whole of this sequence of operations.

12.7 Dry the basin and any residue in an oven at 100±2°C as directed in **12.4**.

12.8 The final weight at this stage shall be called weight B.

12.9 The determination of silicone content shall be made in duplicate.

NOTE 1 — Solvents other than THF may be used, for example, dichloromethane, but complete extraction may take longer (it is virtually complete after one hour using THF) and slightly lower yields may be obtained. Heating by a variably-controlled mantle should be preferred because frothing is less likely to be encountered during extraction. There is also less risk of overheating the extract when removing the solvent at the end of the extraction period.

NOTE 2 — This operation should be conducted in a fume-cupboard because of the inflammability of the solvent and the risk of toxicity attendant upon prolonged inhalation of the vapour. Further, solvent is removed more rapidly and any tendency to frothing is reduced.

NOTE 3 — The volume of hydrofluoric acid used is not critical but should not exceed 5 ml since the efficiency of the chemical reaction is likely to be impaired if the quantity added is large enough to produce inhomogeneity. A polyethylene measuring cylinder should be used for measuring the acid. Considerable care should be exercised in handling hydrofluoric acid because of its corrosive nature. Rubber or plastic gloves and a protective visor should be used. Conducting the subsequent operation in a fume cupboard reduced considerably the extent of exposure to the vapour.

NOTE 4 — Modifications to this procedure are necessary in the determination of silicones in proprietary products containing organo-metallic compounds.

13. RESULTS

13.1 Calculate the silicone content as follows:

$$\text{Silicone (as SiO}_2\text{) percent, by weight} = 100 \times \frac{A - B}{W}$$

where

A = weight in g as determined in **12.5**.

B = weight in g as determined in **12.8**, and

W = weight in g of the test portion of the leather.

14. ACCURACY

14.1 Duplicate determinations shall not differ by more than 5 percent of the mean of the two results.

DETERMINATION OF IRON

[LC : 24]

1. SCOPE

1.1 This method prescribes a procedure for the determination of total iron present in traces for all leathers.

2. OUTLINE OF THE METHOD

2.1 A known quantity of leather is ashed and the amount of iron present is determined colorimetrically in Nessler cylinders by the thiocyanate method.

3. APPARATUS

3.1 Nessler Cylinders — 50 ml capacity.

4. REAGENTS

4.1 Fuming Nitric Acid

4.2 Bromine Water — saturated.

4.3 Concentrated Hydrochloric Acid — *see* IS : 265-1962*.

4.4 Ammonium Hydroxide — 1 : 1 (*v/v*).

4.5 Dilute Hydrochloric Acid — 1 : 1 (*v/v*).

4.6 Dilute Sulphuric Acid — 1 : 1 (*v/v*).

4.7 Potassium Permanganate Solution — approximately 0·1 N.

4.8 Potassium or Ammonium Thiocyanate Solution — Dissolve 10 g of potassium thiocyanate) (or ammonium thiocyanate) in 100 ml of water.

4.9 Stock Iron Solution — Dissolve exactly 0·700 0 g of pure ferrous ammonium sulphate [$FeSO_4$ $(NH_4)_2$ SO_4, $6H_2O$] in 50 ml of water and 20 ml of sulphuric acid, 1 : 4 (*v/v*), and add 0·1 N potassium permanganate solution drop by drop until a faint pink colour is obtained. Adjust the volume to one litre with water. The strength of this solution is ten times that of the standard solution. The keeping quality of this solution is better at this strength. Store this solution and use it when required.

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*Specification for hydrochloric acid (*revised*).

4.10 Standard Iron Solution — Prepare a fresh solution by diluting 10 ml of the stock solution (4·9) to 100 ml with water. Each millilitre of this solution contains 0·000 01 g of iron (as Fe).

5. PROCEDURE

5.1 Ash 5 g of the material, after drying in the oven, in accordance with the method prescribed in LC : 3. Moisten the carbon-free ash with hot water. To this add five drops each of fuming nitric acid and bromine water. Heat slowly until the fumes cease to come out. Evaporate to dryness. Moisten the residue with water and digest it with 5 ml of concentrated hydrochloric acid over a water-bath for a few minutes, dilute with water and filter into a 250-ml beaker. Adjust the volume to approximately 75 ml (by boiling, if necessary) and make it faintly alkaline with ammonium hydroxide. Boil off the excess ammonia gently and allow the precipitated iron and aluminium hydroxides to coagulate.

5.2 Allow the solution to stand for a few minutes until the precipitate settles. Decant the supernatant liquid quickly through an asbestos Gooch filter, washed previously with hot dilute hydrochloric acid, water, ammonium hydroxide and water successively. Wash the precipitate three times with hot water, made faintly alkaline with five drops of dilute ammonium hydroxide per litre. Transfer the precipitate quantitatively to the filter and wash it four times with faintly ammoniacal hot water. Keep the combined filtrate and washings for the determination of copper (*see* LC : 25).

5.3 Dissolve the precipitate on the filter paper by treating it with two lots of 20 ml dilute sulphuric acid and collect the solution. Wash with filter paper four times with dilute sulphuric acid and combine the washings with the solution. Add a few drops of potassium permanganate solution until the solution is slightly pink and transfer it to a 250-ml volumetric flask. Adjust the volume finally to the mark with dilute sulphuric acid.

5.4 Transfer 10 ml of dilute sulphuric acid and 5 ml of potassium thiocyanate solution to each of the two identical 50 ml Nessler cylinders. To one of the Nessler 'cylinders add 10 ml of the made up solution (**5.3**) and make up the volume to 50 ml mark. To the second Nessler cylinder add one millilitre of standard solution and make up the solution to 50 ml mark.

6. RESULTS

6.1 Compare the intensity of the colour produced with the material with the colour produced in the control cylinder.

DETERMINATION OF COPPER

[LC : 25]

1. SCOPE

1.1 This method prescribes a procedure for determining the quantity of total copper for all leathers present in traces.

2. OUTLINE OF THE METHOD

2.1 A known quantity of the leather is ashed and the amount of copper present in the ash is determined colorimetrically in Nessler cylinders by diethyldithiocarbamate method, after eliminating iron.

3. APPARATUS

3.1 Nessler Cylinders — 50 ml capacity.

4. REAGENTS

4.1 Tetrasodium Pyrophosphate Solution — four percent (*w*/*v*).

4.2 Gum Arabic Solution — one percent (*w*/*v*).

4.3 Sodium Diethyldithiocarbamate Solution — 0·2 percent (*w*/*v*). Dissolve the weighed quantity of sodium diethyldithiocarbamate in the requisite amount of copper-free water. Filter and keep in an amber coloured bottle protected from light.

4.4 Stock Solution of Copper — Dissolve 0·392 8 g of copper sulphate crystals ($CuSO_4 \cdot 5\,H_2O$) in water containing a few drops of concentrated sulphuric acid and make up the volume to one litre.

4.5 Standard Solution of Copper — Prepare fresh solution by diluting 10 ml of the stock solution (**4.4**) to 100 ml with water. Each millilitre of this solution contains 0·000 01 g of copper (as Cu).

5. PROCEDURE

5.1 Heat the combined filtrate and washings from **5.2** of LC : 24 until the free ammonia is expelled. Cool and make up to 100 ml.

5.2 Transfer 10 ml of the made up solution to one of the Nessler tubes together with 10 ml of tetrasodium pyrophosphate solution and 5 ml of gum arabic solution. After carefully shaking, add sodium diethyldithiocarbamate solution and make up the volume to 50 ml with water. Carry out a control test as described above using a known quantity of standard copper solution in place of the made up solution.

NOTE 1 — Tetrasodium pyrophosphate solution sequesters manganese, calcium and magnesium salts.

NOTE 2 — Gum arabic solution prevents any separation of the coloured copper complex.

6. RESULTS

6.1 Compare the intensity of the yellow colour produced with the made up solution with that produced in the control test.

DETERMINATION OF PHOSPHORUS

[LC : 26]

(*Adapted from Provisional Method of IUC/15*)

1. SCOPE

1.1 This method prescribes a procedure for determining the total quantity of phosphorus in leather.

2. OUTLINE OF THE METHOD

2.1 The leather is oxidized by a mixture of nitric, sulphuric and perchloric acids to destroy organic matter and convert phosphorus compounds present to orthophosphate. The orthophosphate is determined absorptiometrically as a phospho molybdovandate complex with an absorption maximum of 460 nm.

2.2 Small quantities of biologically derived phosphorus compounds, arising from native hides and skins, are present in leather. Inorganic polymeric phosphates are used as tanning auxiliaries and in the manufacture of other tanning materials. Organic phosphates are used as plasticizers in leather finishing. This recommendation does not differentiate between phosphorus arising from different sources but gives a procedure for determining total phosphorus in leather conveniently expressed in terms of oxide (P_2O_5).

3. APPARATUS

3.1 Analytical Balance with a Sensitivity — of 0·001 g.

3.2 Burettes

3.3 Kjeldahl or Conical Type Fitted with Splash Bulbs — 300 ml flask.

3.4 Measuring Cylinders

3.5 Pipettes

3.6 Spectrophotometer or Absorptiometer with Suitable Filter

3.7 Volumetric Flasks

4. REAGENTS

4.1 Sodium Sulphite — solid.

4.2 Concentrated Nitric Acid — relative density 1·42 (*see* IS : 264-1968*).

4.3 Oxidizing Mixture — a mixture of 70 ml of concentrated sulphuric acid and 230 ml of 60 percent perchloric acid.

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*Specification for nitric acid.

4.4 Dilute Nitric Acid — approximately 3 M.

4.5 Ammonium Molybdate Solution — 5 percent in distilled water (filter after ageing for 24 hours).

4.6 Ammonium Vanadate Solution — 0·25 percent, prepared by dissolving 2·5 g of the salt in 800 ml of hot water containing 20 ml of concentrated nitric acid made up to one litre after cooling.

4.7 Standard Phosphate Solution — containing 1 mg of phosphorus pentoxide (P_2O_5)/ml prepared by dissolving 0·958 5 g of potassium dihydrogen phosphate in distilled water making up to 500 ml.

4.8 Chromium Sulphate — solid.

5. PROCEDURE

5.1 Sample the leathers and prepare a test portion for chemical testing for leather in accordance with IS : 5863-1969*.

5.2 Weigh about 1 g from the test portion to an accuracy of 1 mg into a flask.

5.3 If the leather does not contain chromium, add about 20 mg of chromium sulphate to act as an indicator. Treat the leather with 10 ml of nitric acid in the cold for 10 minutes, then add 15 ml of oxidizing mixture. Place a splash bulb in the neck of the flask. Transfer to a fume cupboard and heat until the mixture boils gently. Continue to heat until the colour changes from green to golden-yellow, remove from the source of heat, cool for a few minutes and add approximately 100 ml of distilled water (*see* Note 1). The first 2 to 3 ml should be added dropwise with shaking. Add about 1 g of sodium sulphite to reduce the chromium and a few glass beads. Boil gently for about 15 minutes to remove sulphur dioxide. Cool, transfer quantitatively to a 100-ml volumetric flask and make up to volume. This is the analytical solution.

5.4 Pipette an aliquot of the analytical solution containing not more than 5 mg of phosphorus pentoxide (P_2O_5) into a 50-ml graduated flask, followed by sufficient distilled water to bring the volume up to 30 ml. Then add, in order, 5 ml of dilute nitric acid, 5 ml of ammonium vanadate and 5 ml of ammonium molybdate, make up to volume with distilled water and mix well. Prepare a blank solution in a similar manner but omitting the ammonium molybdate. Colour development is stable for at least 2 hours. Measure the absorption at 460 mm using the blank prepared from the sample under investigation. Convert to milligram of phosphorus pentoxide (P_2O_5) by the use of a standard calibration curve. For proper colour development the normality of the solution after making up to volume should be between 0·2 to 1·6 N.

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*Methods of sampling for leather.

5.5 The standard calibration curve is prepared by taking aliquots of working standard phosphate solution 0·5 to 5·0 ml in 0·5-ml intervals and developing the colour by the method described in **5.4**. The blank is again prepared by omitting the ammonium molybdate.

NOTE 1 — This operation requires great care and should be carried out in a fume cupboard with the mouth of the flask pointing away from the operator.

NOTE 2 — If leather ash is used for the determination there is no necessity to use nitric acid prior to addition of the oxidizing mixture.

NOTE 3 — Wet oxidation is a procedure requiring care and skill on the part of the analyst who shall observe appropriate safety precautions.

6. CALCULATION

6.1 Calculate phosphorus (as P_2O_5) content by the formula:

$$\text{Phosphorus (as } P_2O_5 \text{), percent by weight} = \frac{10\,000 \times W_1}{VW_2}$$

where

W_1 = weight in g of leather taken,

V = volume in mg of analytical solution taken for colour development, and

W_2 = weight in g of P_2O_5 in the aliquot of analytical solution taken for colour development.

NOTE — The factor 10 000 takes into account the dilutions used in this method.

7. ACCURACY

7.1 Repeatability — Duplicate determination by the same analyst should not differ from each other by more than 0·1 percent calculated on the original mass of leather taken.

7.2 Reproducibility — Differences between laboratories should not exceed 0·2 percent calculated on the original mass of leather taken.

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AMENDMENT NO. 1 JANUARY 1976

TO

IS : 582-1970 METHODS OF CHEMICAL TESTING OF LEATHER

(*First Revision*)

Corrigenda

(*Page* 11, *clause* **3.1.3**):

a) *Line* 4 — Substitute ' Fig. 2 ' *for* ' Fig. 1 '.

b) *Lines* 6 *and* 8 — Substitute ' 10 ml ' *for* ' 5 ml '.

(*Page* 12, *Fig.* 1) — Substitute the figures on pages 2 and 3 of this amendment for the existing Fig. 1.

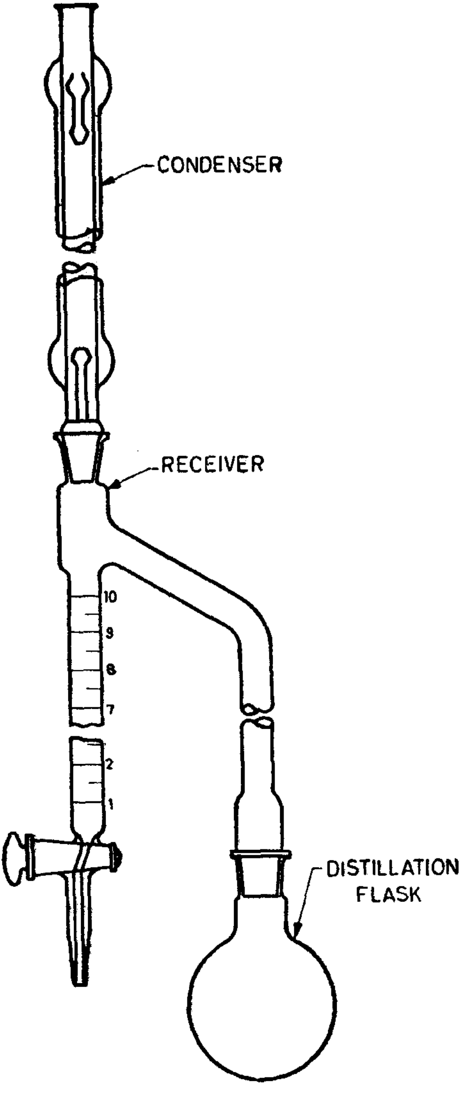
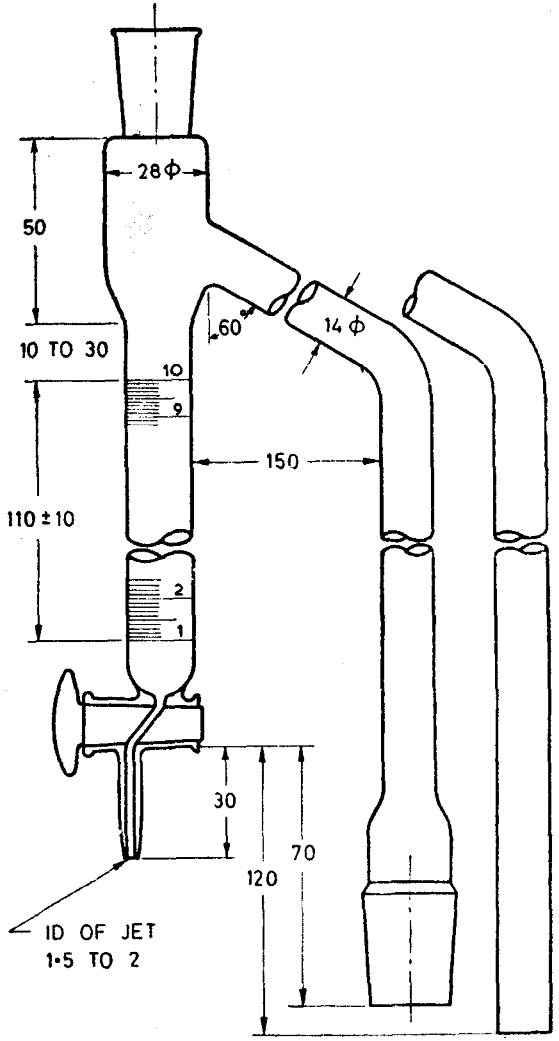


FIG. 1 TYPICAL ASSEMBLY OF APPARATUS FOR DETERMINATION OF MOISTURE



All dimensions in millimetres.

FIG. 2 RECEIVER

(CDC 16)

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